IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of
J. Bednorz et al. : Date: December 15, 1998

Serial No. 08/303,561 : Group Art Unit: 1105

Filed: September 9, 1994 : Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

AFFIDAVIT UNDER 37 C.F.R. 1.132

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Sir:

I, David B. Mitzi, being duly sworn, do hereby depose and state:

That I received a B. S. E. degree in Electrical Engineering/Engineering Physics (1985) from Princeton University and a PhD. degree, in Applied Physics (1990) from Stanford University, California.

That I have worked as a research staff member in Solid State Chemistry at the Thomas Watson Research Center of the International Business Machines Corporation in Yorktown Heights, NY from 1990 to the present.

That I have worked in the fabrication of and characterization of high temperature superconductor and related materials from 1990 to the present.

That I have reviewed the above-identified patent application and that I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and

Muller, which is generally recognized as the first discovery of superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Muller behave in a similar manner, conduct current in a similar manner and have similar magnetic properties.

That once a person of skill in the art knows of a specific transition metal oxide composition which is superconducting above 26°K, such a person of skill in the art, using the techniques described in the above-identified patent application, which includes all knows principles of ceramic fabrication known at the time the application was filed, can make the transition metal oxide compositions encomposed by the claims in the above identified application, without undue experimentation or without requiring ingenuity beyond that expected of a person of skill in the art. This is why the work of Bednorz and Muller was reproduced so quickly after their discovery and why so much additional work was done in this field within a short period of their discovery.

The general principles of ceramic science referred to by Bednorz and Mueller in their patent application can be found in many books and articles published before their discovery. An exemplary list of books describing the general principles of ceramic fabrication are:

1) Introduction to Ceramics, Kingery et al., Second Edition, John Wiley & Sons, 1976, in particular pages 5-20, 269-319, 381-447 and 448-513, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

2) Polar Dielectrics and Their Applications, Burfoot et al., University of California Press, 1979, in particular pages 13-33, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

3) Ceramic Processing Before Firing, Onoda et al., John Wiley & Sons, 1978, the entire book, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

4) Structure, Properties and Preparation of Perovskite-Type Compounds, F.S. Glasso, Pergamon Press, 1969, in particular pages 159-186, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

An exemplary list of articles applying their general principles of ceramic fabrication to the types of materials described in applicants' specification are (these references are cited on applicant's 1449 form submitted August 5, 1987 and in PTO Form 892 in Paper # 20, Examiner's action dated August 8, 1990):

1) Oxygen Defect K_2NiF_4 - Type Oxides: The Compounds La_{2-x} $Sr_x CuO_{4-x/2+\bullet}$, Nguyen et al., Journal of Solid State Chemistry 39, 120-127 (1981).

DBM

2) The Oxygen Defect Perovskite $BaLa_4$ $Cu_5O_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}\,B_{0.5})\,O_3$, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: ____________________

David B. Mitzi

Sworn to before me this 15th day of December, 1998

____

Notary Public

DANIEL P. MORRIS
NOTARY PUBLIC, State of New York
No. 4888676
Qualified in Westchester County
Commission Expires March 16, 1999

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of J. Bednorz et al.	:	Date: December 15, 1998
Serial No. 08/303,561	:	Group Art Unit: 1105
Filed: September 9, 1994	:	Examiner: M. Kopec

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AFFIDAVIT UNDER 37 C.F.R. 1.132

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Sir:

I, Timothy Dinger, being duly sworn, do hereby depose and state:

That I received a B. S. degree in Ceramic Engineering (1981) from New York State College of Ceramics, Alfred University, an M. S. degree (1983) and a PhD. degree (1986), both in Material Science from the University of California at Berkley.

That I have worked as a research staff member in Material Science at the Thomas Watson Research Center of the International Business Machines Corporation in Yorktown Heights, NY from 1986 to the present.

That I have worked in the fabrication of and characterization of high temperature superconductor materials from 1987 to 1991.

That I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Muller, which is generally recognized as the first discovery of

superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Muller behave in a similar way, conduct current in a similar manner and have similar magnetic properties.

That once a person of skill in the art knows of a specific transition metal oxide composition which is superconducting above 26°K, such a person of skill in the art, using the techniques described in the above-identified patent application, which includes all knows principles of ceramic fabrication known at the time the application was filed, can make the transition metal oxide compositions encomposed by the claims in the above identified application, without undue experimentation or without requiring ingenuity beyond that expected of a person of skill in the art. This is why the work of Bednorz and Muller was reproduced so quickly after their discovery and why so much additional work was done in this field within a short period of their discovery.

The general principles of ceramic science referred to by Bednorz and Mueller in their patent application can be found in many books and articles published before their discovery. An exemplary list of books describing the general principles of ceramic fabrication are:

1) Introduction to Ceramics, Kingery et al., Second Edition, John Wiley & Sons, 1976, in particular pages 5-20, 269-319, 381-447 and 448-513, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998

2) Polar Dielectrics and Their Applications, Burfoot et al., University of California Press, 1979, in particular pages 13-33, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

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1) Oxygen Defect K_2NiF_4 - Type Oxides: The Compounds $La_{2-x}Sr_xCuO_{4-x/2+*}$, Nguyen et al., Journal of Solid State Chemistry 39, 120-127 (1981).

2) The Oxygen Defect Perovskite $BaLa_4Cu_5$-$0_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}B_{0.5})0_3$, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: Timothy R. Dinger
Timothy Dinger

Sworn to before me this 16th day of December, 1998

Sandra M. Emma
Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of
J. Bednorz et al. : Date: December 15, 1998

Serial No. 08/303,561 : Group Art Unit: 1105

Filed: September 9, 1994 : Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

AFFIDAVIT UNDER 37 C.F.R. 1.132

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Sir:

I, Chang C. Tsuei, being duly sworn, do hereby depose and state:

That I received a B. S. degree in Mechanical Engineering from National Taiwan University (1960) and M. S. and PhD. degrees, in Material Science (1963, 1966) respectively from California Institute of Technology.

That I have worked as a research staff member and manager in the physics of superconducting, amorphous and structured materials at the Thomas Watson Research Center of the International Business Machines Corporation in Yorktown Heights, New York from 1973 to the present. (See attached Exhibit A for other professional employment history.)

That I have worked in the fabrication of and characterization of high temperature superconductor and related materials from 1973 to the present.

That I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Muller, which is generally recognized as the first discovery of

superconductivity above 26°K and that subsequent developments in this field have been based on this work.

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2) Polar Dielectrics and Their Applications, Burfoot et al., University of California Press, 1979, in particular pages 13-33, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

3) Ceramic Processing Before Firing, Onoda et al., John Wiley & Sons, 1978, the entire book, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

4) Structure, Properties and Preparation of Perovskite-Type Compounds, F.S. Glasso, Pergamon Press, 1969, in particular pages 159-186, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

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2) The Oxygen Defect Perovskite $BaLa_4 Cu_5$-$0_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}\ B_{0.5})$ 0_3,V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: Chang C. Tsuei

Chang C. Tsuei

Sworn to before me this 16th day of December, 1998

Sandra M. Emma

Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

YO987-074BY

CHANG C. TSUEI

Education

California Institute of Technology, M.S. (1963), Ph.D. (1966)

National Taiwan University, B.S. (1960)

Professional Employment

1993 - present - Research Staff Member

1983 - 1993 - Manager, Physics of Structured Materials

1979 - 1983 - Manager, Physics of Amorphous Materials

1974 - 1975 - Acting Manager, Superconductivity

1973 - 1979 - Research Staff Member

Harvard University: 1980 (Summer)

Visiting Scholar in Applied Physics

Stanford University: 1982 (Sept.) - 1983 (April)

Visiting Scholar in Applied Physics

California Institute of Technology

1972 - 1973 - Senior Research Associate in Applied Physics

1969 - 1972 - Senior Research Fellow in Materials Science

1966 - 1969 - Research Fellow in Materials Science

Exhibit A

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: J. Bednorz et al.

Date: December 15, 1998

Serial No. 08/303,561

Group Art Unit: 1105

Filed: September 9, 1994

Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

The Commissioner of Patents and Trademarks
Washington, D.C. 20231

AFFIDAVIT UNDER 37 CFR 1.132

Sir:

I, Thomas M. Shaw, being duly sworn, do hereby depose and state:

I received a B.S. degree in Metallurgy from the University of LIverpool, Liverpool, England and a M.S. and PhD. degree in Materials Science (1981) from the University of California, Berkeley.

I have worked as a postdoctoral researcher in the Material Science Department of Cornell University from 1981-1982. I worked at Rockwell International Science Center in Thousand Oaks, California from 1982-1984 as a ceramic scientist. I have worked as a research staff member in Ceramics Science at the Thomas J. Watson Research

Center of the International Business Machines Corporation in Yorktown Heights, N.Y. from 1984 to the present.

I have worked in the fabrication of and characterization of ceramic materials of various types, including superconductors and related materials from 1984 to the present.

Attached is a resume of my publications. I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Mueller, which is generally recognized as the first discovery of superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Mueller behave in a similar manner, conduct current in a similar manner and have similar magnetic properties.

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work of Bednorz and Mueller was reproduced so quickly after their discovery and why so much additional work was done in this field within a short period of their discovery.

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3) Ceramic Processing Before Firing, Onoda et al., John Wiley & Sons, 1978, the entire book, a copy of which is attached herewith.

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An exemplary list of articles applying their general principles of ceramic fabrication to the types of materials described in applicants' specification are (these references are cited on applicant's 1449 form submitted August 5, 1987 and in PTO Form 892 in Paper # 20, Examiner's action dated August 8, 1990):

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2) The Oxygen Defect Perovskite $BaLa_4 Cu_5$-$0_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5} B_{0.5}) O_3$, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: Thomas M. Shaw
Thomas M. Shaw

Sworn to before me this 14th day of December, 1998.

Sandra M. Emma
Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: J. Bednorz et al.
Date: December 18, 1998

Serial No. 08/303,561
Group Art Unit: 1105

Filed: September 9, 1994
Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

The Commissioner of Patents and Trademarks
Washington, D.C. 20231

AFFIDAVIT UNDER 37 CFR 1.132

Sir:

I, Peter R. Duncombe, being duly sworn, do hereby depose and state:

I received a B.A. degree in Chemistry from the State University of New York at New Paltz, New Paltz, N.Y. and a M.S. degree in Chemical Engineering (1983) from the State University of New York at Buffalo, Buffalo, N.Y.

I have worked as a graduate research assistant in the Chemical Engineering Department of SUNY at Buffalo from 1980-1983. I have worked as a chemical engineer in Ceramics Science at the Thomas J. Watson Research Center of the International Business Machines Corporation in Yorktown Heights, N.Y. from 1984 to the present.

I have worked in the fabrication of and characterization of ceramic materials of various types, including superconductors and related materials from 1984 to the present.

Attached is a resume of my publications (Attachment A).

I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Mueller, which is generally recognized as the first discovery of superconductivity above 26°K and that subsequent developments in this field have been based on this work.

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4) Structure, Properties and Preparation of Perovskite-Type Compounds, F.S. Glasso, Pergamon Press, 1969, in particular pages 159-181, a copy of which is attached herewith.

An exemplary list of articles applying their general principles of ceramic fabrication to the types of materials described in applicants' specification are (these references are cited on applicant's 1449 form submitted August 5, 1987 and in PTO Form 892 in Paper # 20, Examiner's action dated August 8, 1990):

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2) The Oxygen Defect Perovskite $BaLa_4 Cu_5$-$0_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}\ B_{0.5})\ 0_3$, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

I have recorded research notes relating to superconductor oxide (perovskite) compounds in technical notebook IV with entries from November 12, 1987 to June 14, 1988 and in technical notebook V with entries continuing from June 7, 1988 to May 2, 1989. Complete copies of each of these notebooks are attached - Attachment B - Book IV and Attachment C - Book V. Below is a listing of some of the compounds I prepared and recorded in these notebooks according to the teaching as described in the Bednorz and Mueller patent application using the general principles of ceramic science as described in the books and articles listed above.

In Book IV, $Y_1Ba_2Cu_3O_x$ batch C1 pellet pressing, sintering notes and powder processing specifications start on page 2 and continue intermittently to pg. 40 (pg. 13 has superconductive susceptibility curves for pellet 9). Batch C2 $Y_1Ba_2Cu_3O_3$ detailed from pages 14 to 47.

In Book V green phase (Y_2BaCuO_x) microstructural photomicrographs are logged on pages 15-17 with notes continuing to pg. 19. The perovskite superconductor BiSrCaCu oxide ($Bi_{2.15}Sr_{1.68}Ca_{1.7}Cu_20_{8+\delta}$) and related perovskites $Ca_{(2-x)}Sr_xCuOx$ and $Bi_2Sr_2CuO_x$ synthesis notations start and continue through pg. 61 with microstructural photomicrographs.

A series of $Y_1Ba_2Cu_3O_x$ stoichiometric perturbations to study compositional effects on 2nd phase or grain boundary phases and their effect on conductivity (resistivity), sintering behavior etc., continue until the end of the book notes on the page dated May 2, 1989 (page not numbered). These are typical perovskite synthetic procedures, microstructural photomicrographs, powder processing methods, characteristic susceptibility curve(s), sintering behavior and the like. Additional notes may be available in later notebooks.

The undersigned affiant swears further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or patent issuing thereon.

By: Peter R. Duncombe

Peter R. Duncombe

Sworn to before me this 18th day of December, 1998.

Sandra M. Emma

Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

ATTACHMENT A

RESUME 1998

1. Compensation doping of $Ba_{0.7}Sr_{0.3}TiO_3$ thin films
   Copel, M Baniecki, JD Duncombe, PR Kotecki, D
   Laibowitz, R Neumayer, DA Shaw, TM
   APPLIED PHYSICS LETTERS V73 N13 SEP 28 1998 P1832-1834

2. Method for Forming Noble Metal Oxides and Structures Formed Thereof. June 1998.
   Duncombe, P. R. Hummel, J. P. Laibowitz, R. B.
   Neumayer, D. A. Saenger, K. L. Schrott, A. G.
   RC 98A 41575

3. Growth of Bismuth Titanate Films By Chemical Vapor Deposition and Chemical Solution Deposition. March 1998. RC-21124
   Neumayer, D. A. Duncombe, P. R. Laibowitz, R. B.
   Shaw, T. Purtell, R. Grill, A.

4. Dielectric relaxation of $Ba_{0.7}Sr_{0.3}TiO_3$ thin films from 1 mHz to 20 GHz Baniecki, JD
   Laibowitz, RB Shaw, TM Duncombe, PR
   Neumayer, DA Kotecki, DE Shen, H Ma, QY
   APPLIED PHYSICS LETTERS V72 N4 JAN 26 1998 P498-500

5. Contrasting magnetic and structural properties of two La manganites with the same doping levels
   McGuire, T.R. Duncombe, P.R. Gong, G.Q. Gupta, A. Li, X.W. Pickart, S.J. Crow, M.L.
   J. Appl. Phys. (USA) Vol.83, No.11 1 June 1998 P7076-8

6. Effects of Annealing Conditions on Charge Loss Mechanisms in MOCVD $(Ba_{0.7},Sr_{0.3})TiO_3$ Thin Film Capacitors.
   Baniecki, J.D., Laibowitz, RB Shaw, TM Duncombe, PR Saenger, KL Cabral C
   Kotecki, DE , Shen, H , Lian, J., Ma, QY

7. Low Operating Voltage and High Mobility Field Effect Transistors Comproising Pentacene and Relatively High Dielectric Constant Insulators RC21233(94806) 7/17/98
   Dimitrakopoulos, CD Purushothaman S , Kymissis J. Callegari A. , Neumayer DA, Duncombe PR, Laibowitz RB, Shaw JM

8. Maximum Magnetorsistance in Granular Manganite/Insulator System close to Percolation Threshold PACS 10/06/98
   DK Petrov, L Krusin-Elbaum, JZ Sun, C Feild, & PR Duncombe

9. Magnetorsistance and Hall Effect of Chromium Dioxide Epitaxial Thin Films
   X.W. Li, A. Gupta, T.R. McGuire, P.R. Duncombe, Gang Xiao

10. Progress Report on High-k dielectric material: amorphous BST from solgel (09/98)
    P. Andry, D. Neumayer, P. Duncombe, C. Dimitrakopoulos, F. Libsch, A. Grill, R. Wisnieff

RC21352(96175)2Dec1998

InfoGate from The IBM Total Information Retrieval Center

SEND MAIN MENU OTHER OPTIONS

INCOMPLETE

Personal Inventor History

Name:Duncombe, P.R. Serial:155139 Loc:RES YORKTOWN
Patent Pts:36 TDB Pts:1 Total Pts:37 Plateau Lvl:3
Plateau Date:10/24/98 File Update:11/02/98
Awards Due:None

Title: NOVEL METAL ALKOXYALKOXIDECARBOXYLATES AND USE TO FORM FILMS
06/17/98 Opened as Discl YO8980231 Status:Filed
06/22/98 Discl Review Action:File
1 09/04/98 Filed as Docket YO998254 in US Rating: 2 Pts:3
Co-inventors: Neumayer, D.A.

Title: SELECTIVE GROWTH OF FERROMAGNETIC FILMS FOR MAGNETIC MEMORY, STORAGE-BASED DEVICES, AND OTHER DEVICES
06/17/98 Opened as Discl YO8980225 Status:Filed
06/29/98 Discl Review Action:File
4 10/15/98 Filed as Docket YO998268 in US Rating: 2 Pts:3
Co-inventors: Guha, S. Gupta, A. Bojarczuk, N.A. Karasinski, J.M.

Title: BEOL DECOUPLING CAPACITOR MATERIALS
01/28/98 Opened as Discl YO8980024 in US Status:Opened
06/24/98 Discl Review Action:File
Co-inventors: Rosenberg, R. Ning, T.H. Shaw, T.M. Edelstein, D.C. Neumayer, D.A. Laibowitz, R.B.

3 "FABRICATION OF STRONTIUM Bismuth Tantalate/Bismuth Titanate Multilayer FERROELECTRIC
Title: FERROELECTRIC THIN FILM STRUCTURES
10/01/97 Opened as Discl YO8970512 in US Status:Opened
09/16/98 Discl Review Action:File
Co-inventors: Shaw, T.M. Neumayer, D.A. Laibowitz, R.B.
2 10/30/98 SENT TO COUNSEL (L. Schiws)

Title: CAPACITORS WITH AMORPHOUS DIELECTRICS AND IMPROVED DIELECTRIC PROPERTIES MADE USING SILICON SURFACES AS ELECTRODES
06/06/97 Opened as Discl YO8970261 in US Status:Opened
Co-inventors: Shaw, T.M. Neumayer, D.A. Laibowitz, R.B.

Title: FABRICATION OF THIN FILM FIELD EFFECT TRANSISTOR COMPRISING AN ORGANIC SEMICONDUCTOR AND CHEMICAL SOLUTION DEPOSITED METAL OXIDE
03/25/97 Opened as Discl YO8970113 Status:Filed
03/25/97 Discl Review Action:File
03/25/97 Filed as Docket YO997083 in US Rating: 2 Pts:3
6 03/24/98 Filed as Docket YO997083 in JA Rating: 2
03/16/98 Filed as Docket YO997083 in TA Rating: 2
03/12/98 Filed as Docket YO997083 in KO Rating: 2
04/24/98 Last Office Action
Co-inventors: Purushothaman, S. Dimitrakopoulos, C.D. Furman, B.K. Neumayer, D.A. Laibowitz, R.B.

Title: NOVEL ALKOXYALKOXIDES AND USE TO FORM FILMS
10/30/96 Opened as Discl YO8960411 Status:Filed
03/10/97 Discl Review Action:File
5 01/30/98 Filed as Docket YO997069 in US Rating: 2 Pts:3
Co-inventors: Neumayer, D.A.

Title: THIN-FILM FIELD-EFFECT TRANSISTOR WITH ORGANIC SEMICONDUCTOR REQUIRING LOW OPERATING VOLTAGES
09/11/96 Opened as Discl YO8960358 Status:Filed
03/04/97 Discl Review Action:File
(7) 03/25/97 Filed as Docket YO997057 in US Rating: 2 Pts:3
03/12/98 Filed as Docket YO997057 in KO Rating: 2
04/10/98 Last Office Action
Co-inventors: Purushothaman, S. Dimitrakopoulos, C.D. Furman, B.K. Neumayer, D.A. Laibowitz, R.B.

X **Title:** HIGH DIELECTRIC CONSTANT, BARIUM LANTHANUM TITANATE THIN FILM CAPACITORS FOR RANDOM ACCESS
06/20/96 Opened as Discl YO8960255 in US Status:Opened
Co-inventors: Gupta, A. Shaw, T.M. Laibowitz, R.B.

Title: METHOD FOR FORMING NOBLE METAL OXIDES AND STRUCTURES FORMED THEREOF
10/30/95 Opened as Discl YO8950450 Status:Filed
11/12/96 Discl Review Action:File
11/05/97 Filed as Docket YO996239 in US Rating: 2 Pts:3
(8) 10/20/98 Filed as Docket YO996239 in JA Rating: 2
07/30/98 Filed as Docket YO996239 in TA Rating: 2
Co-inventors: Schrott, A.G. Saenger, K.L. Hummel, J.P. Neumayer, D.A. Laibowitz, R.B.

Title: PEROXIDE ETCHANT PROCESS FOR PEROVSKITE-TYPE OXIDES
10/23/95 Opened as Discl YO8950434 Status:Filed
08/08/97 Discl Review Action:File
(9) 04/08/98 Filed as Docket YO997256 in US Rating: 2 Pts:3
Co-inventors: Rosenberg, R. Cooper, E.I. Laibowitz, R.B.

Title: RF TRANSPONDER FOR METALLIC SURFACES
08/02/95 Opened as Discl YO8950329 in US Status:Opened
Co-inventors: Afzali-ardakani, A. Feild, C.A. Duan, D.W. Brady, M.J. Moskowitz, P.A.

Title: METHOD FOR CLEANING THE SURFACE OF A DIELETRIC
09/06/95 Opened as Discl FI8950292 Status:Filed
09/06/95 Sent to Evaluator
02/05/96 Evaluated Action:Search
04/19/96 Discl Review Action:File
12/06/96 Filed as Docket FI996047 in US Rating: 2 Pts:3
11/29/97 Filed as Docket FI996047 in KO Rating: 2
05/26/97 Filed as Docket FI996047 in TA Rating: 2
06/11/98 Last Office Action
Co-inventors: Kotecki, D.E. Wildman, H.S. Yu, C. Natzle, W. Laibowitz, R.B.

Title: NANO PHASE FABRICATION OF COPPER-GLASS CERAMIC COMPOSITE VIAS IN CORDIERITE SUBSTRATES
10/05/92 Opened as Discl YO8920907 in US Status:Published
10/08/92 Sent to Evaluator
12/17/92 Discl Review Action:Publish
01/06/93 Mailed to Tech Discl Bulletin 09/02/93 Published Pts:1
Co-inventors: Kang, S.K. Shaw, T.M. Brady, M.J.

Title: METHOD OF SINTERING ALUMINUM NITRODE
11/06/92 Opened as Discl FI8920668 in US Status:Closed
11/06/92 Sent to Evaluator
12/18/92 Closed
Co-inventors: Takamori, T. Shinde, S.L.

Title: METHOD OF SINTERING ALUMINUM NITRIDE

11/06/92 Opened as Discl [illegible]8920667 in US Status:Closed
11/06/92 Sent to Evaluator
12/18/92 Closed
Co-inventors: Takamori, T. Shinde, S.L.

Title: ALUMINUM NITRIDE BODY AND METHOD FOR FORMING SAID BODY UTILIZING A VITREOUS SINTERING ADDITIVE
08/13/92 Opened as Discl FI8920525 Status:Filed
08/17/92 Sent to Evaluator
09/29/92 Evaluated Action:Search
12/23/92 Discl Review Action:File
05/10/95 Filed as Docket FI992168B in US Rating: 2 Pts:3
05/28/96 Issued as Patent 5520878 in US
Co-inventors: Takamori, T. Shinde, S.L.

Title: ALUMINUM NITRIDE BODY AND METHOD FOR FORMING SAID BODY UTILIZING A VITREOUS SINTERING ADDITIVE
08/13/92 Opened as Discl FI8920525 Status:Filed
08/17/92 Sent to Evaluator
09/29/92 Evaluated Action:Search
12/23/92 Discl Review Action:File
12/22/93 Filed as Docket FI992168A in US Rating: 2 Pts:3
01/09/96 Issued as Patent 5482903 in US
Co-inventors: Takamori, T. Shinde, S.L.

Title: GOLD DOPING OF YBA2CU3O7-8 AS A MEANS OF INCREASING TRANSPORT CRITICAL CURRENT DENSITY
02/12/92 Opened as Discl YO8920161 in US Status:Closed
02/14/92 Sent to Evaluator
05/15/92 Closed
Co-inventors: Daeumling, M. Shaw, T.M.

Title: PROCESS FOR PRODUCING CERAMIC CIRCUIT STRUCTURES HAVING CONDUCTIVE VIAS
07/19/89 Opened as Discl YO8890552 Status:Filed
07/25/89 Sent to Evaluator
08/10/89 Evaluated Action:Search
07/30/90 Discl Review Action:File
12/17/92 Filed as Docket YO990091B in US Rating: 2 Pts:3
08/16/94 Issued as Patent 5337475 in US
Co-inventors: Vallabhaneni, R.V. Giess, E.A. Farooq, S. Cooper, E.I. Kim, Y.H. Vanhise, J.A. Aoude, F.Y. Muller-landau, F. Shaw, R.R. Walker, G.F. Rita, R.A. Neisser, M.O. Park, J.M. Shaw, T.M. Brownlow, J.M. Kim, J. Knickerbocker, S.H.

Title: VIA PASTE COMPOSITIONS AND USE THEREOF TO FORM CONDUCTIVE VIAS IN CIRCUITIZED CERAMIC SUBSTRATES
07/19/89 Opened as Discl YO8890552 Status:Filed
07/25/89 Sent to Evaluator
08/10/89 Evaluated Action:Search
07/30/90 Discl Review Action:File
03/20/91 Filed as Docket YO990091A in US Rating: 2 Pts:3
02/01/94 Issued as Patent 5283104 in US
Co-inventors: Vallabhaneni, R.V. Giess, E.A. Farooq, S. Cooper, E.I. Kim, Y.H. Vanhise, J.A. Aoude, F.Y. Muller-landau, F. Shaw, R.R. Walker, G.F. Rita, R.A. Neisser, M.O. Park, J.M. Shaw, T.M. Brownlow, J.M. Kim, J. Knickerbocker, S.H.

Call your award coordinator, IPL department, or T/L 826-2680 for help.

SEND | MAIN MENU | OTHER OPTIONS

NAME: Duncombe Peter R [redacted] Emp. Ser: 1551[redacted] Date: 10/23/95

- T.R. McGuire, A. Gupta, P.R. Duncombe, M. Rupp, J.Z. Sun, R.B. Laibowitz, W.J. Gallagher & G. Xiao "Magnetoresistance and Magnetic Properies of $(La_{1-x})MnO_{3-\delta}$ Thin Films" 3M Conf. Proc: 4/96
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- J.Z. Sun, L. Krusin-Elbaum, A. Gupta, G. Xiao, P.R. Duncombe, W.J. Gallagher & S. P. Parkin "Magneto-Transport in Doped Manganate Perovkites" 3M conference 11/12-15/96 Atlanta, Georgia
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- P.R. Duncombe. S.L. Shinde, & T. Takamori "Aluminum Nitride Body Utilizing A Vitreous Sintering Additive" US05482903 1/9/96 (EF Plaque)
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- Ali Afzali-Ardakani, Mike Brady, Dah-Weih Duan, Peter Duncombe, Chris Feild, and Paul Moskowitz "RF Transponder for Metallic Surfaces" Docket#:YO895-0329 submitted: 8/2/95
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- E.I. Cooper, P.R. Duncombe, R.B. Laibowitz, "Peroxide Etchant Process for Titanate Dielectrics" Docket: YO895-0434 rated file; in prep.
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- A. Grill, R. Laibowitz, D. Beach, D. Neumayer & P.R. Duncombe "Effect of Base Electrode on the Crystallization & Electrical Properties of PLT" IBM RC 20402 (90185) 3/5/96
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- C.D. Dimitrakopoulos, P.R. Duncombe, B.K. Furman, R.B. Laibowitz, D. Neumayer, S. Purushothaman, J. Shaw "Field Effect Transistor for Low Voltage Operation" Disclosure YO896-0358 rated file: 9/11/96
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IBM Commitments: To Win To Execute To Teamwork

ATTACHMENT B

T01001

IBM

Technical Notebook

Book IV

User's Initials and Last Name:
P DUNCOMBE

Employee Serial:	Date of First Entry:	Date of Last Entry:
155139		

11/12/87 6/88

Security Classification:

MORAR

11/12

70/30 - 25-25 ? } C1GEP2 - ~12:30 start

123 basis

3.094 ?	0.574	0.179		4.07	"63.9" ~~34.6~~ !?
	1.458	0.455	0.760		
~4 hrs 3.047	0.515	0.158		5.65	88.7 better
≤~1.5% loss	1.308	0.401	0.539		p1 → 83.4

11/12

$SrTiO_3$ - ST3 → 32 hrs ST2 pcs. → 48

11/13

$SrTiO_3$ ⇒ ST3 → cooled in morning see book III, pg A7

4.024	0.510 ✓	0.240 ✓		~ 5.01	"1.04(2) % dense"
no wght loss	1.295	0.610	0.803	same	Sally

~48 hrs (+ cooling 3 mornings, stepwise) sintering pellet

Cutting record
"

start 0.425 (0)
+ 0.060 Δ - saw (0.015) = 0.045 ~ 1.14 mm w/ flattening ~1 mm ✓
.485 (Δ 0 ↳ 0.042 (1.08 mm) ok
0.060
0.545 (0.045 Δ reading; actual ⇒ 0.052 → 1.32 mm

55 .0465 1.18 mm

0.0523

bottom (0.6 - 0.69) not flat 1.52 mm

10/13
2

PRE
ST4 - some deformity on 1 side (a slice worth) — IN w/ final piece of ST2 @ 4:55

4.178 0.584 ~0.287 3.316 68.9
1.483 0.729 1.26 ~average

will remove monday morn ∴ ~6-6 ~~SAT~~, 6SAT-6SUN, ~63+ hours projected

10/16
4.169 0.510 ~0.250 4.98 1.035 <CONSISTENT>
(0.2 %) 1.295 0.635 0.837

G1 ISO-26,000 UNL-3300
4.01 0.578 0.248 ~~0.542~~ 3.96
1.468 0.630 ~~0.992~~
1.066

density est. (figure 65%) => 5.785 RANGE (5.37 - 6.27)

C1ZT1
10/17 3300/26,000 EXTREMELY SHINY, flat surfaced, ~~with~~ sharp cornered pellet
3.105 0.566 0.193(4) 3.90 61.2%
1.438 0.490 0.796

G1 16hrs
3.985 0.578 0.250 (no change) 3.738 (lost 0.5% density)

10:18 IN hot furnace, packed T_c - 520C

	T_c	T_s	T_{SET}	
20	977	745	971	to 'push' ΔT
21	↓	838	✓	
22		898		
23		935		23½ => 951
24		949	956	~SET-PT.
2:23				"OFF" for slo-cool (first stepped to 840)
2:54	435			

10/17

C1 z f 1 ⇒ pellet multiply cracked as if organic residue vaporized, evidence of vapor transport to support plate, etc. ~~are~~ Not

2.925 Δ0.18
5.5%

9.79 - 3.105 ⇒ 6.685

10/18
G1 - post 4.044 split in 4 pieces (seemingly on cooling)

G2 4.1	0.579	0.253		3.75 ✓	pellet slightly disfigured, but ok.
	1.471	0.643	1.093		
33					
	4.155				
	0.510	0.220		5.64	about expected density
	1.295	0.559	0.736		
D.D.1 Pre					
3.10	0.5765"	0.191"			
S.1.4	0.513	0.165		5.61	88.2
	1.303	0.419	0.559		

4
11/24

IBM Technical Notebook

Thermdyne Tube furnace set-up specs -
thermcouple : dia. ~0.255 length 20" + USED 23"
Set-up complete w/ plug in jacks, ext. wire, S couples.

11/30 Analytical Submission

C1 -	0.75 g	$Y_{0.02}Ba_{0.38}Cu_{0.6}$	Y, Ba, Cu
C2 -	1.1	Y_2O_3	Y, trace 99%
C3 -	2.0	"BaO"	Ba,
C4 -	1.0	TiO_2	Ti, trace
C5 -	2.0	$SrTiO_3$ pre	Sr, Ti, trace
C6 -	1.0	↓ post mill	↓
C7	2.0	DRC 123 .	Y, Ba, Cu
C8		DD 123	Y, Ba, Cu
C9		off comp 2:11	

'New' ~~20~~ 20.25 g GRINDING CHARGE of $SrTiO_3$ IN MILL (3:10) off (4:17)
O_2, compressed AIR, CO_2 cylinders obtained w/ regulators
Ar

YIELD ⇒ 20.4g ∴ MUST BE some from old batch OR ZrO_2
COMBINED W/ OLD PWDR → 23 g of milled powder

12/2 C1 batch 45.6 GRAMS left
39.56 g (~6 g kept for files
10.5
29.5 left for pellets
~10 for grinding charge TFE/Toluene

New BOTTLES ORDERED, NO TEFLON AVAILABLE, - approx - 60 hrs total

$SrTiO_3$ pellets ⇒ 10-10 (24) hrs Tues down N4 ∴ 2-2am Wed (~12?) / ~12-12 Thurs → (24)

ST~~4~~5, ST6 - start 10 A.M. 12/~~9~~, numerous interruptions due to furnace malfunctions, out 12:00 P.M. 12/10

ST5 edge chips 1 side OK otherwise 21+150.

*' 4.08	0.285	0.584 0.52			
(4.01)	0.237 0.602	0.520 1.321	0.825	4.94	1.027

ST6 long chip during iso pressing in 3/4 side, must do

4.128	0.586 ~~0.520~~	0.286 ~~0.237~~			
4.15	0.513 1.303	0.249 0.632(5)	0.843	4.92	1.023

*' bisecting crack

950C RUN

GREEN	C1P9 3.108	0.577 1.466	0.185 0.470	0.793	3.92	61.5
POST	3.1 no loss	0.514 1.306	0.161 0.409	0.548	5.66	88.9%
GREEN	DRC2 3.204	0.579 1.471	0.177 0.450	0.765	4.19	65.8
POST	3.2 no loss	0.551 1.400	0.165 0.419	0.645	4.96	77.9

12/7 pellets not in best shape after 150 at 26000

975C RUN

	C1P10 3.090 3.056	0.574 1.459	0.185 0.470	0.785	3.99	61.9
POST	1% loss	0.508 1.288	0.157 0.399	0.520	5.88	92.3 → PROBABLY 93 +3.4%
	*DRC3 3.318	0.579 1.471	0.181 0.460	0.782	4.24	66.6% ~
POST	crack still apparent, but holding 3.293 0.75% loss	0.547 1.389	0.168 0.427	0.647	5.09	79.9 + 2%

C1P10 Pynometer 91.8 → 92 ∴ MOSTLY closed porosity (95% on pure density basis)

* cracked in 1/2, but holding. Will go w/ & see if it heats.
To temp @ 4:05 ⇒ 2 HRS 6:05 start ramp down)
9:00 A.M 12/8 start cooling, OX NOT FLOWING WHEN ARRIVED, THOUGH COULD HAVE HAD BACK PRESSURE

12-8

100% E/G mix ⇒ new wght calc.

(4.0 g) E basis (transfered to jar for physical mixing)

92.0913g/mM cation ∴ 4.0/92.0913 = 0.0434 mM

0.0434 mM is BASIS for mix of 0.7 mM Eutectic

0.0434/0.7 = 0.0620 mM total ∴ 0.3 mM 211

0.3 (0.0620) = 0.0186 mM (94.6725g/mM 211) = 1.761g 211

1.761 g 211
4.0 g E
5.76(1) g mix

tare 0.83 5.76 / 5.68 recovered / .08g loss on mixing 5.53 after grinding (slight loss on transfer)

1 pellet pressed ⇒ EG1 ⇒ to temp 12/10 @ 3:40-45 PROJ OUT TO BE 5:45

2.57 0.580 0.153 3.88 ~~60.9~~ ⇒ 74%?
1.473 0.389 0.663

Rel. density calc 0.3 (6.00) + 0.7 (4.9) ⇒ 5.23 approx theoretical
EMPIRICAL D's

2.543 (1% wght loss) ~0.611 0.161 2.825
1.55 0.480 0.90

Restarted for overwrite RUN

12/9

IBM Technical Notebook

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

Ba_2Cu_5O

SAMPLE I.D. BaCuO eutectic   DATE ______
SOURCE D.Goland   OPERATOR PRD
TOTAL WEIGHT 18.855 g.   OUTGASSING CONDITIONS ______
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 14.794 g.   ADDED VOLUME, V_A ____ cc
CELL HOLDER VOLUME, V_c ____ cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

DATA

	R=3.687 RUN 1	3.688 RUN 2	RUN 3
P_2	18.362	18.488 (7)	
P_3	4.980	5.013	
V_p	3.023 cc	3.034 cc	cc
DENSITY	4.89 g/cc	4.88 g/cc	g/cc

± 0.24 (5%)
4.65 ↔ 5.13
{ previous error } { lost contents }

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III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

Bulk SAMPLE I.D. α-N_2O_2 DATE 12-9-87   α-S
SOURCE Sumitomo   OPERATOR PRD
POST 9.212 / 4.062 / 5.15
TOTAL WEIGHT 12.606 PRE   OUTGASSING CONDITIONS ______   13.867
TARE WEIGHT 4.06 g.   4.061
SAMPLE WEIGHT 8.546 g.   ADDED VOLUME, V_A 85.52 cc   9.806
CELL HOLDER VOLUME, V_c 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

POST - Sumitomo

	R=3.637	R=3.638
P_2	18.501	18.464
P_3	5.087	5.079
V_p	2.419	2.395
D	4.05	(+2.3%) 4.09 (+3.3%)

DATA

	PRE (archive) RUN 1	POST RUN 2 R=3.549	RUN 3 R=3.549
P_2	18.320	18.502	18.534
P_3	5.078	5.213	5.222
V_p	– cc	1.296 cc	cc
DENSITY	4.15 g/cc	3.96 g/cc	g/cc

-2.608

Loss 3.96 / 5%

Right On

16

III. DENSITY WORKSHEET

best guess - 5.9
STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 211   DATE 12-9-87   RUN 2
SOURCE PRD   OPERATOR PRD
TOTAL WEIGHT 19.662 g.   OUTGASSING CONDITIONS ______   20.477
TARE WEIGHT 4.061 g.   4.061
SAMPLE WEIGHT 15.601 g.   ADDED VOLUME, V_A ____ cc   16.416
CELL HOLDER VOLUME, V_c ____ cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

DATA

	R=3.65 RUN 1	R=3.651 RUN 4	R=3.663 RUN 2	R=3.664
P_2	18.557	18.568	18.504	18.529
P_3	5.084	5.085	5.052	5.098
V_p	2.578 cc	cc	2.736 cc	2.748
DENSITY	6.05 g/cc	g/cc	6.080 g/cc	5.97

± 0.3
(5.75 - 6.35)*
*probably ≤ 6.05 (an underestimate) [illegible]

Ave Run {2
6.00

16

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 123   DATE 12-9-87
SOURCE DRC   OPERATOR PRD
TOTAL WEIGHT 21.026 g.   OUTGASSING CONDITIONS ______
TARE WEIGHT 4.063 g.
SAMPLE WEIGHT 16.963 g.   ADDED VOLUME, V_A 34.85 cc
CELL HOLDER VOLUME, V_c 85.52 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

DATA

	R=3.6625 RUN 1	R=3.628 RUN 2	RUN 3
P_2	18.598	18.596	
P_3	5.078	5.078	
V_p	2.73 cc	cc	cc
DENSITY	6.21 g/cc	g/cc	g/cc

16

12/9

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 123-3μm DATE 12-9
SOURCE DIMOS OPERATOR PRD
TOTAL WEIGHT 19.700 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 15.639 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_C 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R=3.646 R→3.646 ~~R=3.6??~~

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.603	18.561	~~18.5??~~
P_3	5.103	5.091	~~5.0??~~
V_p	2.523 cc	cc	cc
DENSITY	6.199 g/cc	g/cc	g/cc

No purge

16

III. DENSITY WORKSHEET

STEREOPYCNOMETER Run 2
TRUE POWDER DENSITY

SAMPLE I.D. 123 DATE 12-9
SOURCE C1 OPERATOR PRD
TOTAL WEIGHT 22.944 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 18.883 g. ADDED VOLUME, V_A 34.85 cc
CELL HOLDER VOLUME, V_C 85.52 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R→ 3.642

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.508		
P_3	5.014		
V_p	3.082 cc	cc	cc
DENSITY	6.13 g/cc	g/cc	g/cc

16

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 123 DATE 12-9-87
SOURCE C1 - 3μm OPERATOR PRD
TOTAL WEIGHT 14.459 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.063 g.
SAMPLE WEIGHT 10.396 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_C 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R=3.58 R=3.58

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.679	18.645	
P_3	5.218	5.208	
V_p	1.703 cc	cc	cc
DENSITY	6.10 g/cc	6.105 g/cc	g/cc

NOT BACK TO ZERO

16

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. C1P10 DATE 12-10-87
SOURCE C1-975248 OPERATOR PRD
TOTAL WEIGHT 6.613 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 2.552 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_C 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R- 3485 3485

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.148	18.182	
P_3	5.208	5.217	
V_p	0.4355 cc	cc	cc
DENSITY	5.859 g/cc	g/cc	g/cc

on 6.13 → 95.6
6.37 → 91.8 (92)

16

7.668 × 5875

12/10

IBM Technical Notebook

Powders for Analysis ⟹ Never entered @ conf. time 12/11, Sensitivities NOT enough. Need to increase by 10X at least.

	Species	Description
~~C1~~ -	Y_2O_3	left exposed to air
	TiO_2	
C1	YBa_2Cu_3 ($Y_1Ba_2Cu_3$) ↓	
DD1	↓	
DRC	↓	
PII	↓	off comp
EI	$Y_{0.02}Ba_{0.38}Cu_{0.6}$	off comp

Table I - Precision[1] of Metals determined by ICP in $La_{1.8}Sr_{0.2}CuO_4$ and $YBa_2Cu_3O_7$ Thin Films.

Element	x^2	S.D.	R.S.D. (%)
La	1.80	0.08	4.64
Sr	0.20	0.01	5.52
Cu	1.00	0.14	3.52
Y	1.00	0.05	5.60
Ba	2.04	0.07	3.43
Cu	3.00	0.11	3.67

[1]Based on 7 determinations

[2]Calculated atomic ratios

For 123

Y (0.333) ⟹ ± 0.019 0.314 - 0.352

Ba (0.667) ⟹ ± 0.023 0.644 - 0.690

Cu (1.00) ⟹ ± 0.0367 0.963 - 1.0367

The above understood and witnessed by Date and by Date

Theoretical wgt % calcs.

			Anal 1	
TiO_2 ⇒	47.90/79.8988	→ 59.95	57.3	ERROR reported.
$SrCO_3$ ⇒	87.62/147.62935	→ 59.35		NOT ANALYZED
$BaCO_3$ ⇒	137.34/197.34935	→ 69.59(2)		
BaO ⇒		89.566	88.9	99.26 !

$SrTiO_3$ ⇒ Sr ⇒ 47.74(5) M.W. 183.5182
Ti ⇒ 26.10(1)

C5-	22.2	Ti	85.05% (15% poor)
	49.4	Sr	"3.48% rich"
C6	24.2	Ti	92.72 (7.3% poor)
	50.6	Sr	"5.98%" Rich

86.5
↕
92.5

12/14 both well shaped pellets

C1P11 - 150 26

3.673	0.574	0.215		4.03	63.3
	1.458	0.546	0.9116		

92+ (5/14) final from T.SHAW

C1fp(#)? 15026

3.058	0.561	0.206		3.606	56.6 as usual
	1.437	0.523	0.848		

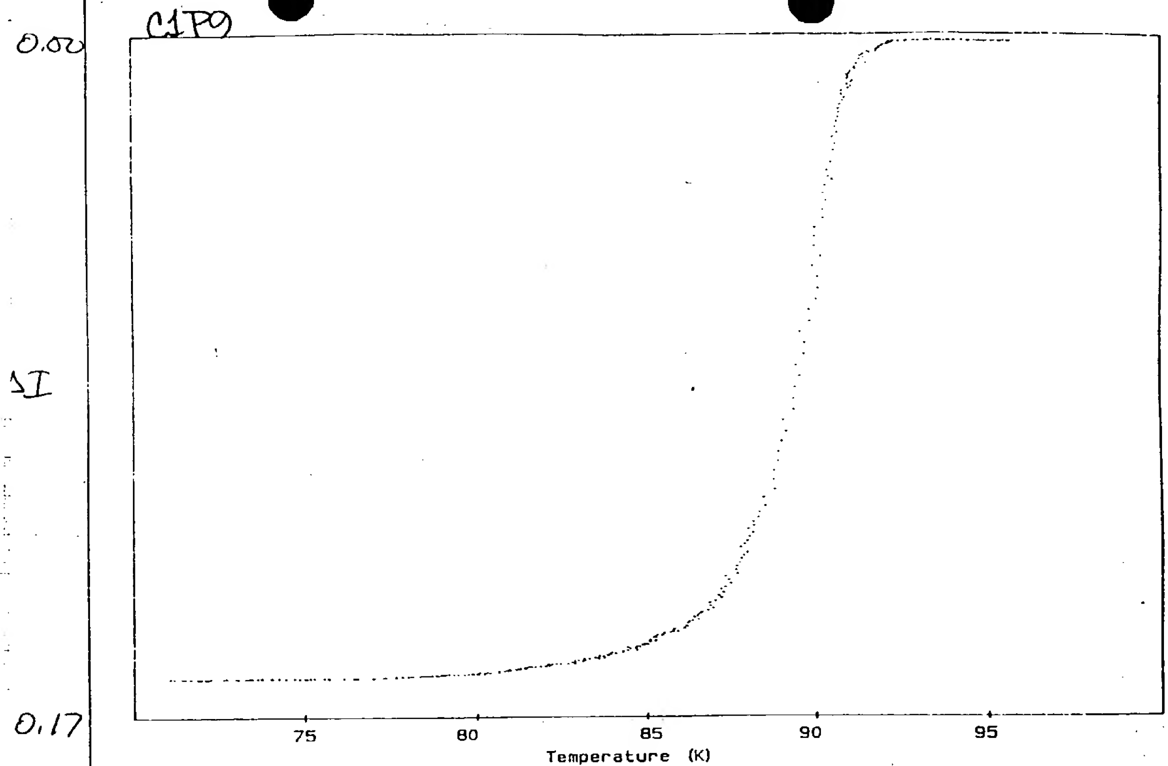
- final microstructure full of liquid, bimodal g.s. & cracking
- no final density recorded

12/16 pellets in furnace from 12/14 in O_2 purge.
To temp (10°C/min ramp from RT) @ 10:50 A.M.
Low (leading) side undershoot 974, high (downside) overshoot 978.
Stable variation 974-976 ✓

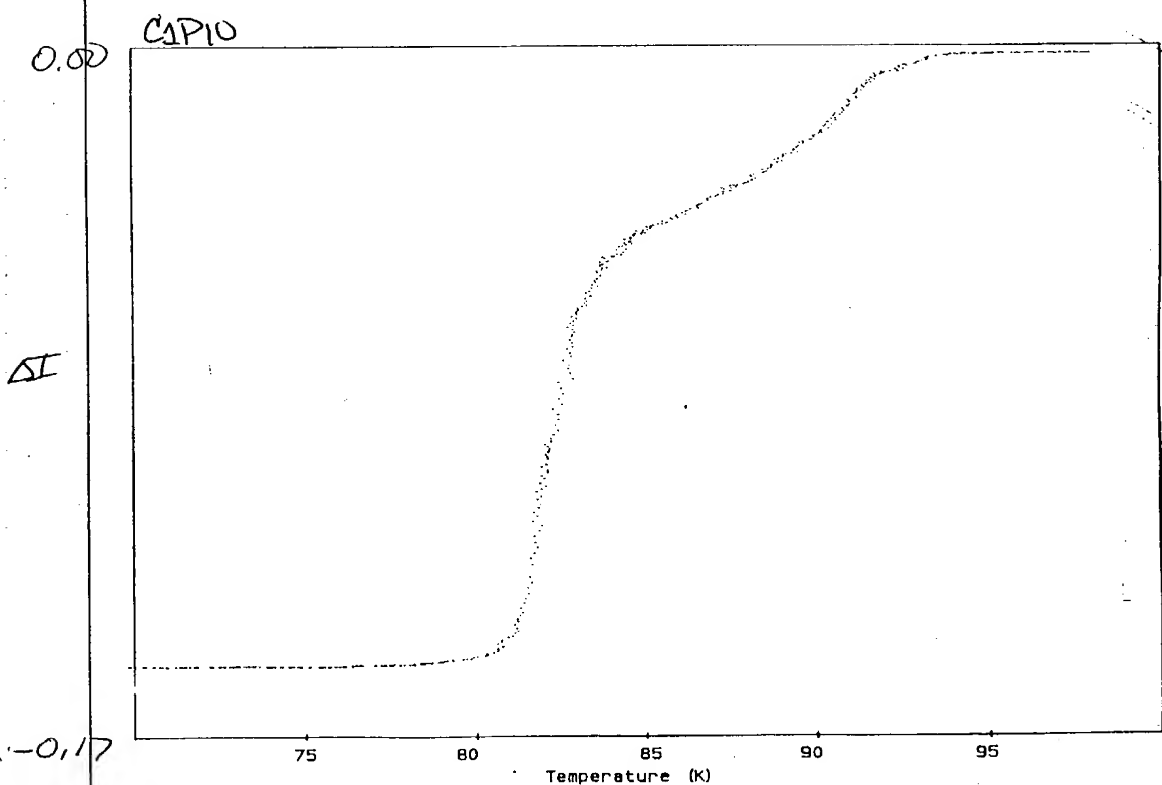
start ramp down ~~12:50~~ 1:00 p.m. (to 600C where soak for 48 hours)

{Dif coef: $2 \times 10^{-15}\, m^2/s^{-1} \times 2 \times 10^{-15} \frac{m^2}{s} \times \frac{ft^2}{(0.3048\,m)^2} = 2.153 \times 10^{-14}$ ft^2/s }

C1P9


0.02
ΔI
0.17
75
80
85
90
95
Temperature (K)

C1P10


0.02
ΔI
-0.17
75
80
85
90
95
Temperature (K)

C2 Batch → $Y_1Ba_2Cu_3O_7$ 200g

From C1 batch calc, (pg. 54 Book III) 72 Book II)

	wt. Frac.	Purity	Mult.	final
Y_2O_3 ⇒	17.1535 ⇒	17.1707 ⇒	x2	34.34
BaO_x ⇒	46.5934	100 ⇒	x2	93.1868

$BaCO_3$ conversion: 93.1868 $\frac{197.35}{153.34}$ = 119.932(3) ÷ 0.99 ⇒ 121.14(4)

CuO ⇒ 36.25(81) ⇒ 36.2893(4) ⇒ x2 72.57(9)

O.K. everything is Ba rich by analysis, so why not ~~not~~ correct ⇒ 119.93
Apply 1/2. +.606
120.54

$BaCO_3$

tare: 279.67
+ 120.54
400.21 won't read, but will tare

Reads: 120.57(4-6) was 4/5

CuO

tare: 0.86/7
Reads 72.58 transfered quant. tare to zero w/ paper

Y_2O_3 transferal quant

34.34/5 paper weighs ~0.1 when checked due to static glove charge, but after glove/charge removal 0.00. Think OK since cal-up before glove use (not more than 0.3% error)

Expected total weight } 227.46 g dry

BaO - 5.72 g/cc $BaCO_3$ - 4.43 Y_2O_3 - 5.01 CuO - 6.3-6.49

∴ if bumping occurs w/ selective loss, $BaCO_3$ should preferentially be lost if not ~~well~~ uniformly suspended.

Except for 1 bump (0.06 g recovered) ⇒ very smooth, uneventful preparation. Placed in drying oven for weekend drying. (oven cleaned before use also)

12/21 After breaking up cake and re-baking under vac @ 70C for 3+ hrs.

~~Crux~~ #1 transferal   ideally want 75 per crux

166.67 → 0.97
tare 86.21
80.46 g
−.01 "recovery"

totals
80.46
77.74
68.29
226.49 expected 227.46 (99.57%)

0.3 g recovered on ~~[illegible]~~ breaking cake.

CRUX 2 172.72
94.98
77.74
+.03 "recovery"

226.79 total 99.7%
227.46

CRUX 3 173.46
105.17 "white"
68.29

Rxn. Run 1 ⇒ N₂ on @ 4:10 pm 320 ramp to 940C, 450 cool ramp
14 hrs + 3 up + 2 down = 20 hrs total

12/22 [illegible]

{12/21}

12/17-18 CENTURR $SrTiO_3$ RUN

Δ 500 psi

2100 - 1700 psi Ar usage 16 HR SOAK @ 1800 w/ 16.7 up/400 down ramp

Running Si-Christy run Prog. 05

∴ for 12/21 ⇒ ⊗ $SrTiO_3$ RUN 24 hrs ⇒ 1000 psi
16
40 hrs ⇒ 1500 psi max permissable

Set for 36 ⇒ RAMP STARTED @ ~1:25 p.m. 12/21
3 HRS to temp
4:25
36 hours SOAK
40:25
4.25
1600 psi @ 300C RAMP up. 44.5 hours total should be O.K

(3 HRS ⇒ 1000) ⇒ 15,000 projected usage.

12/22 9:00 AM
19.3 soak hours left ∴ Δt ⇒ 16.7 + 3 ⇒ 19.7 {(16 - 11250) psi ⇒ 4,750
∴ 241.1 psi/hr. 19.3 + 4.25 = 23.55 (241.1 psi/hr) = 5,680
11250 - 5680 = REMAINDER of 5,572 psi } could run longer if rate remains constant

6:00 pm
11,250 - 9000 ⇒ 2,250/(16.7 - 10.2) = 2250/6.5 = 346.2 !
346.2 (10.2 + 4.25) = ≈5,000 psi + (9000) = 4,000 to spare ✓

12-22

C2 RXN SEEMS GOOD, NO APPARENT LIQUID, LARGE SHRINKAGE NO VISIBLE GREEN, GOOD BLACK COLOR, BEFORE UNLOADING - ~~Post wght.~~

CRUX #1
initial 166.97
tare 86.21
80.76

100% EXPECTED RXN wght.

80.76 + (80.76 × (- 0.1182)) = 71.214

loss calc.

227.46 theoretical powder

$\frac{120.54}{227.46} = 0.52994$ wght % $BaCO_3$

$\frac{153.34}{197.35}(0.52994) = 0.41176$

$\Delta = 0.52994 - 0.41176 = 0.1182$ % total

WRONG?

CRUX #2
initial 172.72
94.98
77.74

as above = 68.551

crux #3
173.46
105.17
68.29

as above = 60.218

199.983 total / 0.997 = 200.58 ✓✓ OK.

Actual yields - 14 HR RXN @ 940C

		total wght initial tare	ABOVE EXPECTED WGHT,	% RXN
CRUX #2 2.27g wght loss	170.45 ~~94.98~~ ~~75.47~~	75.47	6.919 (~~9.489~~)	24.7
CRUX #1 2.05g wght loss	169.92 86.21 78.71	78.71	7.496 (9.546)	21.5
CRUX #3 1.55g wght loss	171.91 105.17 66.74	66.74	6.522 (8.022)	19.2

CONSISTENT

21.8% aver. } O.K.

Recheck of wght loss calc.

Ba 137.34
O 15.9994
153.3394 ~~153.34~~
~153.34

Ba 137.34
3 Os 47.9982
1C 12.01115
197.34935
~197.35

$\frac{153.34}{197.35} = 0.776995\ (120.54) = 93.6589997\ \Delta 26.881\ g$

26.881 g / 3 cruc. = ~8.96 g / crucible ~ correct

Individual wght inquiries during grinding 12/29

crux 3 66.72 unloaded

105.17 tare
171.82 loaded
171.91 previously
.09 g loss = 0.135%
66.74

169.38
171.82
−2.44 loss
+ −1.55
3.99

crux ~~2~~ 1 86.20 tare (0.19/20)
78.75 load (−0.02) 78.73
78.69 gram after grinding
.06 g loss > 0.076% loss
164.85
78.65 0.09 g loss = 0.115% los

161.68
164.85
−3.17 loss
+ −2.27
5.44 total to date

crux ~~1~~ 2 75.49/8 unloaded
94.99/8 tare
75.46 load (pre crux)

170.44 loaded.
75.46 0.03 loss

167.18
170.44
−3.26 loss
2.05
5.31

Samples were incompletely converted, as wght loss indicated.
Top was black, but went through a transition of greens progressly
lower in crucible.
Grnd powder was a dull forest green. Crux 1 slightly dark than 2 & 3.
All had white hard agglomerates (presumably $BaCO_3$).
Tops inhibited oxygen flow throughout crucible. No tops used
for second RXN. Heat treatment

12/29 New losses consistent w/ crux loading. Conversion now up
to 70.8 ~ 71%. ~~Will~~ regrind and ~~refire~~

initial weighing — loss

crux 2
unloaded 95.02
ground 72.10

crux 1 86.27 ✓
grd. 75.40

crux 3 105.19
grd pot 64.15

crux 1 (3 fuel load) 192.24 / 86.29/1 } 188.21 −4.03 22.82 expected 26.807 (85.1%)

199.53 / crux 2 95.01 } 195.48 −4.05

Reground, to 1 crucible

∴ (202.47 − 199.73) = 2.74 g loss (1.35%)

286.00
86.27
199.73 of 202.47 to start (before grd)

123 Wght Loss Summary (by run & crucible)

Crucible #	Initial	Post (1)	Post (2)		Post (3)	Crucible
1	~~80~~.76	78.71	75.48		102.0	1 & 3
2	77.74	75.47	72.20		100.47	2
3	68.29	66.74	64.21		202.47	
	226.79	220.92	211.89			
theoret	227.46					

final grd into 1 crucible: pre 202.47
grnd post 199.73
loss 2.74g (1.35% ⇒ 1g spill of sieved powdr)

Was 85% reacted before this run.
Total loss so far slightly less than 24.32 g / ~~2~~27.46 (88.5–85%)
Expect less than, but approx. 3.0 g loss for complete rxn.
0.52994 % $BaCO_3$ { (0.776995 % of $BaCO_3$ is BaO)
Look for 283 g total upon cooling!

1/5
initial wght. ~~286.00~~ 285.91
post ~~284.17~~
001.83 g

~~199.73~~ 199.61 initial
197.59 unloaded
2.14

1/4 $SrTiO_3$ synthesis II   references book III pgs. 77, A3

TiO_2 - 79.8988 g/M

$SrCO_3$ - 147.6235

$SrTiO_3$ - 183.5182

SrO - 103.6194

Ti - 47.90   in $SrTiO_3$ 26.1009

Sr - 87.62   in   47.7446

Take transferred amount to shaker jar ($SrCO_3$) as basis for TiO_2 addition

~~201.66 g tare~~ $\frac{48.50}{147.6235}$ (0.32854 moles) x 79.8988 g/M TiO_2 = 26.2498
= 26.2547

~~110.86~~

251.07
202.56
48.50

251.07
26.25
277.32

/.999 =

251.07
26.2763
277.35 target
46

actual 277.35/6

COMPS SCRIPT A1 dated 87/12/02 14:32:25 ..... Page 1

theoretical

Date: 2 December 1987, 13:24:31 EST
From: PLECHAT at YKTVMZ
To: PRD

The laboratory results on your samples are:

Ti consistently low 26.1

Sr consistently high 47.7

# C1	Y 0.03	Ba 0.68	Cu O X	Cu=1,	ICP
# C2	Y ... 78.1 % (W/W)				
# C3	Ba ... 88.9 %				
# C4	Ti ... 57.3 %		/ error due to static electr. during weighing of sample!/		
# C5	Ti ... 22.2 % Sr ... 49.4 %				
# C6	Ti ... 24.2 % Sr ... 50.6 %				
# C7	Y 0.34	Ba 0.71	Cu O X	Cu=1	
# C8	Y 0.34	Ba 0.71	Cu O X		
# C9	Y 2.37	Ba 1.10	Cu O X		

MHP

Date: 21 October 1987, 10:45:18 EDT
From: PLECHAT at YKTVMZ
To: PRD

The laboratory results on your samples are:

# C1	Y 0.35	Ba 0.72	Cu O X	Cu=1,	ICP
# C1f	Y 0.33	Ba 0.70	Cu O X		
# C5	Y 2.21	Ba 1.06	Cu O X		

Other results to follow from Olson,
MHP
Note: I have produced a light green compound from 123 with the formula: $Y_{12}Ba_3Cu\,O_X$. If interested get in touch with me.

4/21
283.17
TARE 86.61
196.56

initial 197.59
196.56
1.03 g lost during grinding

POST
196.56
194.16
-2.40

Lost another 2.4 g. Must be totally converted @ this point.

UNIaxial - 7,000/0.371 = 18,870 PSI
0.126 0.6H 2.53 4.19 65.8
0.320 1.55 0.604

C2P1 green
not final dens. data

C2P2 ⇒ Will leave notes on pwdr later. too busy. Still see
liq. on crucible however, disheartening.
↳ 1 mill 4.2 μm PSD 10-1 ~ flat dist.

green 27 isopressed
3.59 0.580 0.194 4.274 67.1% (high vs C1)
1.473 0.493 0.84

C2P3 - (Green) mill 2 2.53 μm ave., much better behaved pellet

3.55 0.576 0.210 3.96 62.2% (good agreement w/ C1)
1.463 0.533 0.896

C2P2 - removed @ 600 °C ⇒ 20°/up to 800, 10°/to 975, 20° down

3.59 0.554 0.186 4.89 76.8% ! terrible
1.407 0.472 0.734
slightly higher due to pellet attention

1/13
C2P3 3.59 0.51~~7~~ 0.185 5.57 87.4%
1.313 0.47 0.636

~88

 1/13

IBM Technical Notebook

1 transient 996 peak

C2P4 3,775 UNI/26,000 ISO 988 2HRS/600 over

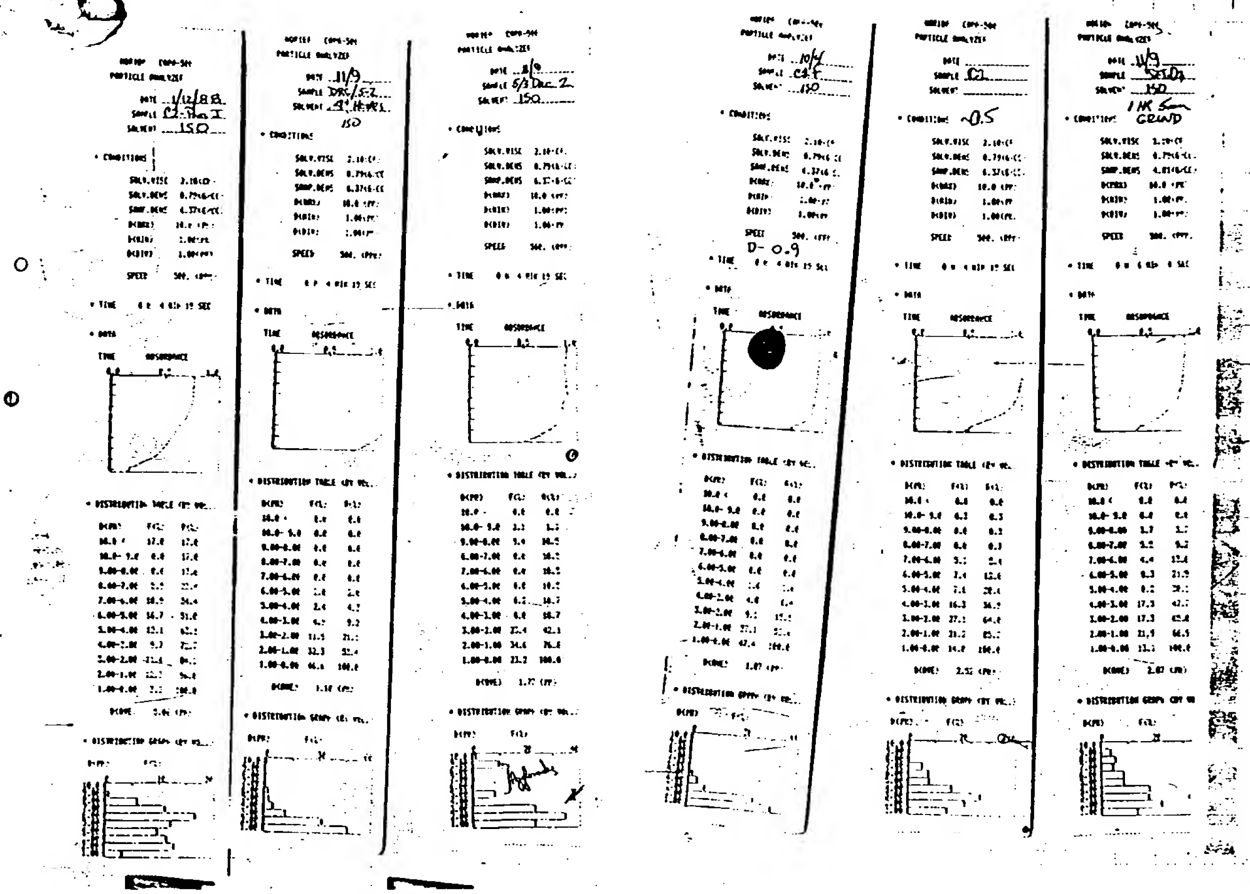
3.50/1	0.576	0.206		3.987	62.6%	CONSISTENT
	1.463	0.523	0.879			
1/14 3.49	0.515	0.180		5.68	89.2	
	1.308	0.457	0.614			

C2P5 3775/24— 990

3.18	0.577	0.199		3.74	58.7%
	1.466	0.505	0.85		
3.15	0.496	0.168		5.92	92.9
	1.26	0.427	0.532		

pellet has stress cracking and inhomo microstructure with large grain interior and peripheral eggshell of small grains.

PSD SHEETS



see pg 27 for REST

HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE DRC
SOLVENT

* CONDITIONS 1.5 HRS

SPEED 500. (RPM)

* TIME 0 H 4 MIN 15 SEC

* DATA

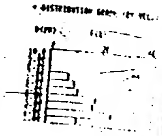
TIME ABSORBANCE

* DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
30.0 <	0.0	0.0
30.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	7.5	7.5
6.00-5.00	[illegible]	[illegible]
5.00-4.00	11.2	[illegible]
4.00-3.00	18.1	[illegible]
3.00-2.00	17.2	[illegible]
2.00-1.00	24.1	[illegible]
1.00-0.00	11.6	100.0

D(MED): 2.00 (µm)

* DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 11/4
SAMPLE 3 HRS DRC 5 am
SOLVENT 150

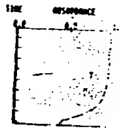
* CONDITIONS

SPEED 500. (RPM)

* TIME 0 H 4 MIN 15 SEC

* DATA

TIME ABSORBANCE

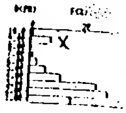


* DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
30.0 <	0.0	0.0
30.0- 9.0	0.4	0.4
9.00-8.00	7.5	[illegible]
8.00-7.00	0.0	[illegible]
7.00-6.00	1.8	9.1
6.00-5.00	2.5	[illegible]
5.00-4.00	5.9	17.5
4.00-3.00	11.6	[illegible]
3.00-2.00	25.2	54.4
2.00-1.00	29.7	84.0
1.00-0.00	16.0	100.0

D(MED) 2.17 (µm)

* DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 9/25
SAMPLE DRC-A
SOLVENT 150

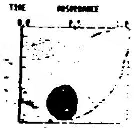
* CONDITIONS

SPEED 500. (RPM)

* TIME 0 H 4 MIN 15 SEC

* DATA 102

TIME ABSORBANCE

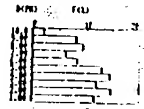


* DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
30.0 <	0.0	0.0
30.0- 9.0	2.0	[illegible]
9.00-8.00	0.1	[illegible]
8.00-7.00	9.3	15.4
7.00-6.00	[illegible]	21.6
6.00-5.00	[illegible]	[illegible]
5.00-4.00	13.1	47.0
4.00-3.00	14.5	61.6
3.00-2.00	12.1	73.6
2.00-1.00	14.7	[illegible]
1.00-0.00	11.7	100.0

D(MED) 3.00 (µm)

* DISTRIBUTION GRAPH (BY VOL.)



402

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 12/2
SAMPLE SCTiD3-92
SOLVENT 150

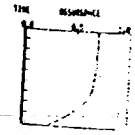
* CONDITIONS

SPEED 500. (RPM)

* TIME 0 H 6 MIN 0 SEC

* DATA

TIME ABSORBANCE

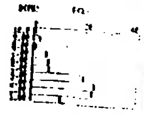


* DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
30.0 <	0.0	0.0
30.0- 9.0	0.4	0.4
9.00-8.00	2.2	2.6
8.00-7.00	[illegible]	4.8
7.00-6.00	[illegible]	9.1
6.00-5.00	6.7	[illegible]
5.00-4.00	6.7	21.6
4.00-3.00	30.0	41.6
3.00-2.00	[illegible]	[illegible]
2.00-1.00	[illegible]	[illegible]
1.00-0.00	[illegible]	100.0

D(MED): 2.67 (µm)

* DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 11/12
SAMPLE CL-6HS/2
SOLVENT 150

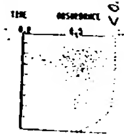
* CONDITIONS

SPEED 500. (RPM)

* TIME 0 H 4 MIN 15 SEC

* DATA 40 < 0

TIME ABSORBANCE



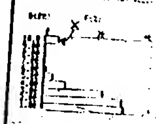
* DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
30.0 <	0.0	0.0
30.0- 9.0	[illegible]	[illegible]
9.00-8.00	0.0	[illegible]
8.00-7.00	0.0	[illegible]
7.00-6.00	0.0	[illegible]
6.00-5.00	0.0	[illegible]
5.00-4.00	3.2	[illegible]
4.00-3.00	[illegible]	[illegible]
3.00-2.00	22.5	[illegible]
2.00-1.00	29.7	[illegible]
1.00-0.00	30.3	100.0

60%

D(MED) 1.44 (µm)

* DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 11/19/87
SAMPLE CL-9 HRS 92
SOLVENT 150

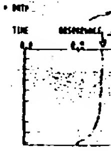
* CONDITIONS

SPEED 500. (RPM)

* TIME 0 H 4 MIN 15 SEC

* DATA

TIME ABSORBANCE



* DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
30.0 <	0.0	0.0
30.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	3.5	3.5
7.00-6.00	0.7	4.2
6.00-5.00	3.4	7.6
5.00-4.00	5.5	13.2
4.00-3.00	10.0	23.1
3.00-2.00	13.9	37.0
2.00-1.00	25.2	[illegible]
1.00-0.00	33.7	100.0

6

D(MED): 1.56 (µm)

* DISTRIBUTION GRAPH (BY VOL.)

Date and sign every entry. Ha[illegible] [illegible]ssibly important entry witnessed. Submit an In[illegible] [illegible]losure of anything possibly new and inve[illegible].

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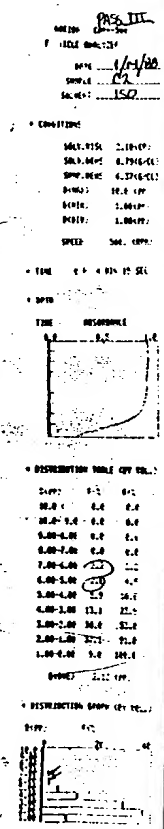

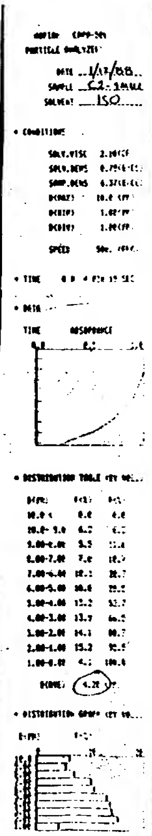

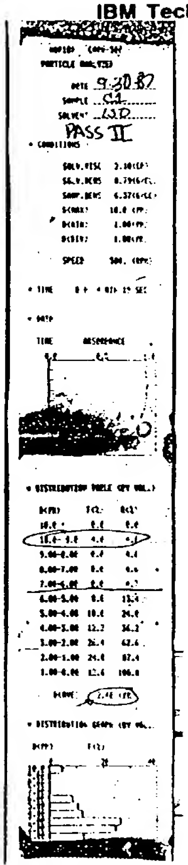

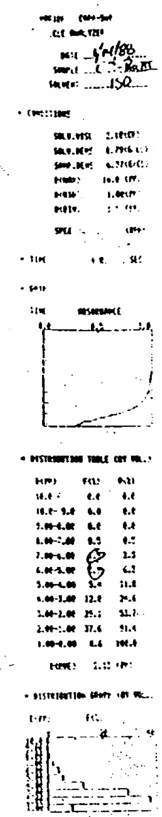

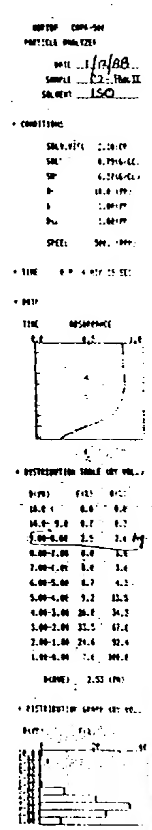

The above understood and witnessed by | Date | and by | Date

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IBM Technical Notebook

28 1/19 (18, 17, 16, 15 holiday)

NOTE ⇒ C1 powder

C1P12, 13, 14, 15 3.775/26.5 —

C1P12

Green 3.04	0.574	0.178		3.92	61.5%
	1.478	0.452	0.7755		
3.01	0.506	0.153		5.966	93.66%
	1.285	0.389	0.504(5)		

C1P13

Green 3.00	0.574	0.175		3.93(4)	61.8%
	1.478	0.444(5)	0.7626		
2.97	0.506	0.150		6.01	94.35
	1.285	0.381	0.494		

C1P14 (*)

Green 2.89	0.574	0.169		3.92(7)	61.6%
	1.478	0.429	0.736		

C1P15 (*)

Green 3.05	0.575	0.179		4.00	62.8%
	1.460(9)	0.455	0.762(9)		

(*) NO DATA ON final pellets - Tom took

The above understood and witnessed by ____________ Date ____________ and by ____________ Date

1/19 Runs in furnace as: all ramps 10C/min

HOOD | 2 HRS : SINTER TIME

Furnace 1 (12) (13) Furnace 2 (14) (15)

1/19 A.M.

4:10 P.M. 4:25 P.M.

to temp (975 C)

6:10 P.M.

RAMP down to 600C SOAK

1/20 1:49 P.M.

RAMP down to RT

CHECK 2:22 (270C)

Pellet thickness experiment DD mill powder 3275/26,000

DT2.0

2.04	0.575	0.119		4.03	63.3 %
2.01	1.460(5)	0.302	0.506		
	0.507	0.100		6.09	
	1.288	0.254	0.33		95.6

DT 1.5

1.54	0.575	0.090		4.01	62.95 %
1.51	1.460(5	0.229	0.384		
	0.509	0.075		6.04	94.8
	1.293	0.190(5)	0.250		

DT 1.0 X

1.09	0.575	0.065		3.95	62.0 %
	1.460(5)	0.165	0.276		

Cutting Calculations for C1P12, 13

0.~~5~~ 99
0.06 / 3 blade thickness + 0.0?
0.~~09~~ 93/3 = 0.0~~3~~ 310

C1P12 (0.025) 5 = 0.125
(↓) 6 = 0.150 ← O.K. from micrometer

use 2 cuts { no 'paralleling' 0.~~0~~50
0.040
0.11/3 = 0.037+0.015 = 0.0517
in from edge

1 cut MADE, BUT PELLET HAS CRACK.

(0.025) 6 = 0.150
0.040
0.11/3 = 0.037 + 0.015 = 0.052

1/21

DT 1.751 in furnace/no green data (5°C RAMP to try to eliminate SINTER-CRACKING)

DT 1.75(2)

1.88 0.575 0.111 3.98 62.5 Never RUN
1.4605 0.282 0.472

Stereopycnometer   1/27/88 {25/26 supply, miller repairs}
28
29, 2/01

See sheets

Data Points (Multiples)

NOTE pwdr: 97.3 − 95.8

	D_m		D_p
"83"	82.95	DRC, DDP12	95.8
"86"	86.4	JP2b2, C1P3, C1fp2	92.2
"89"	89.3	C1fp1, C1p4, C1P7	89.56
"91"	91.3	C1P1, C1p5, C1P8	91.9

← Δ86.4−89.3 = Δ3%

Single Point trends ⊗

87.5	87.5	JP1	83	NOT clear, ~~could~~ seems to be closed
77		C2P2	95.4	DEFINitely would open δ
"93"	93	DDP13	86.6	indicates closure

⊗ small volumes yield low D values for closed porosity.

3500/26,000

2/02 Sintering : Porosity Inquiry C1 & C2 @ 975

{ 10°/min Ramp from RT, 2 hour soak, 10°/ ⇒ to RT no O_2 equilibration. In order from left to right in crux,

C1P16

3.03	0.575	0.178		4.00	62.8	GREEN polished
	1.460(5)	0.452	0.757			
3.00	0.509	0.152		5.92	92.9	
	1.293	0.386	0.507			

C1P17

3.26	0.575	0.191		4.01	62.9(5)	GREEN
	1.460(5)	0.485	0.812(5)			
3.22	0.508	0.164		5.914	92.8	
	1.290	0.4166	0.544(5)			

C2P6

	0.575	0.194		3.82(6)	60.0	GREEN
3.16	1.460(5)	0.493	0.826			
3.11	0.497	0.160		6.12	96.1	
	1.262	0.406	0.507(8)			

C2P7 chip ⊛

	0.575	0.199		3.79	59.5 ⊛	GREEN polished
3.21	1.460(5)	0.505(5)	0.847			
3.16	0.497	0.164		6.065	95.2	
	1.262	0.4166	0.521			

C2P7 good & dense, but exterior cracking due to oxygen penetration. Will quick cool by opening furnace. Quench.

IIICM

C2-8	3.08	0.573	0.191		3.82	59.97 ⇒ ~60%
		1.155	0.485	0.806		comparable to previous
	3.06(5)	0.523	0.154		5.53	see
		1.328	0.399	0.553		86.8
C1-18	3.07	0.578	0.178		4.01	62.95 ~ 63%
		1.468	0.452	0.765		comparable to previous
	3.03	0.497	0.158		6.04	94.8
		1.262	0.401	0.5016		

2/12

HP-4 green 5,000/27,000

13.98	0.947	~0.301		4.02(5)	63.2
	2.405	0.764(5)	3.473		

Citing C2-8 dry
1st 0.50

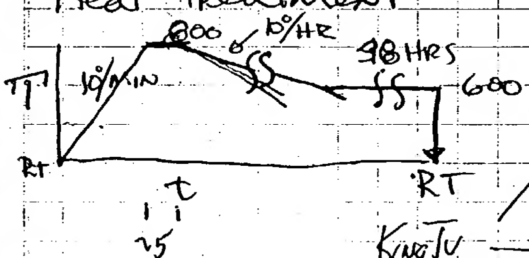
2/15

Boat Spots → positioning I.D.

(1 2 3 4 5 6 7)
45MW

1. } large grain material } C1 3/4
2. }

Heat Treatment



3 } C2 std. w/ surface cracking (outer)
4 } inner surfaces should be o.k.

5. Good C2 top slice - no surface cracking
6. middle slice of 3/4 (w/ chip)
7. bottom slice of C2-5 above.

START: 4:50 PM 2/12 → 6:15 7:00 start ramp down 20 hrs to soak point
~~[illegible]~~ -3:00 ~ 30 HRS

QUENCH! 2:50 2/15

2/17 3500/26750

C2-9	3.09	0.575	0.193		3.76	59.0%
		1.460(5)	0.490	0.821		
	3.06	0.510	0.168			85.5 !
		1.295	0.427	0.562		
C2-10	3.06	0.575	0.191		3.77	59.2%
		1.460(5)	0.485	0.812(5)		
	3.025 3	0.501	0.164		5.77	~~90.66~~
		1.272(5)	0.417	~~1.29~~ 0.530		89.6

Furnace O_2 purge > 1 HR @ 29 (32) C 12:10 PM, ∴ 945/10 = 94.5 mins / 60 MIN/HR = 1.575 HRS OR 1 hr 34.5 mins (1:45 START SINTER)

1:45 - 2:15 (1/2 hr sinter) w/ quench.

C2-11	3.02	0.575	0.188		3.775	59.3% O.K.
		1.460(5)	0.477(5)	0.80		
	2.98	0.505	0.159		5.71	89.64 ~90
		1.283	0.404	0.522		

D ≤ 4 µm   D ≤ 6 µm   D ≤ 6-7   D ≤ 9

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/18
SAMPLE C2-III.OI
SOLVENT 150
D > 0.83

• CONDITIONS

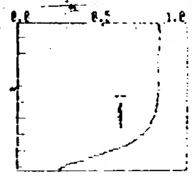
SOLV.VISC	2.16 (CP)
SOLV.DENS	0.79 (G/CC)
SAMP.DENS	6.37 (G/CC)
D(MAX)	10.0 (µm)
D(MIN)	1.00 (µm)
D(DIV)	1.00 (µm)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 19 SEC

• DATE

TIME   ABSORBANCE

0.0   0.5   1.0



• DISTRIBUTION TABLE (BY VOL.)

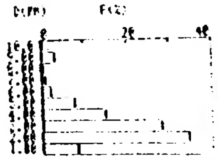
D(µm)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	2.9	2.9
8.00-7.00	0.3	3.2
7.00-6.00	0.7	3.9
6.00-5.00	1.8	5.7
5.00-4.00	7.8	13.5
4.00-3.00	15.1	28.6
3.00-2.00	28.2	56.8
2.00-1.00	34.7	91.5
1.00-0.00	8.5	100.0

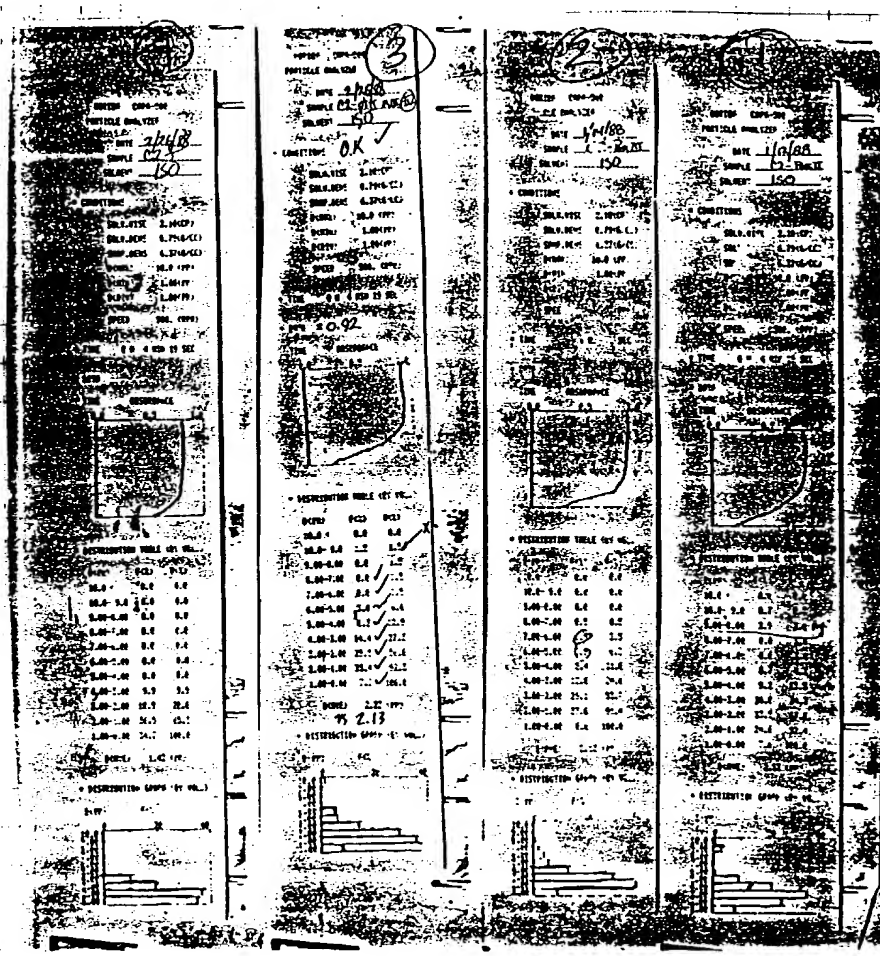
D(AVE): 2.24 (µm)

• DISTRIBUTION GRAPH (BY VOL.)

D(µm)   F(%)

0   20   40





C2 PSDS
1) C2 MILL PASS II
2) C2 ↓ PASS III of 1/2 of ①
③ C2 MILL PASS IV⊛ of other 1/2 of ①
④ fines from ①, ②, ③ III
⊛ 3rd milling was ineffective due to clogged bag & powder charged channels.

 $SrTiO_3$ 3/4 Synthesis

(see Book III page 77 for work-up)
: NO comp ⇒ 698, 39's

Prep:

tare 206.15

$SrCO_3$ 50.00 g desired

256.15

256.15/6 actual wght

0.0 Δ

TiO_2 27.062 desired

283.212 desired

283.22 actual wgh

+0.01 Δ

+0.01 scale replace

~0.0 Δ net ✓

1 hr+ mixing

TRANSfer

tare 89.20

166.23 final wght

77.03+

77.062 expected

-0.03 g's Δ 0.04

185.56 total prelim. wght w/ top

19.33 g top

theoretical expected 151.36 w/out top

19.33

120.69 w/ top

3/5 150.97

14.87 + 62.16 = 77.03 ~ correct

Ramp @ 700C/HR to 1450C ⇒ to temp ~3:25

0.39/150.97 (0.258% loss)

GROUND yield ⇒ 61.39/62.16 ⇒ 98.8%

~1% grinding loss

Clean X-RAY. Milled 1 HOUR

SYN PROJECT COMPLETE
3/5

Date and sign every entry. Have ... entry witnessed. Submit an Inve... ...ly important ...ure of anything possibly new and inven...

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3/4/88

C2 pellets C2p12-15: batch 1 mill III
C2p16-17 2 "IV"

3700/27000

C2p12 3/21 → page 44	~~3.075~~ (3.08)	0.572 1.453	0.191 0.485	0.804	3.825	60.0
C2p13 3/21 → page 44	3.02 (3.02)	0.573 1.455	0.188 0.477(5)	0.794	3.803(5)	59.7
C2p14 page 47	3.11	0.574 1.458	0.192(3) 0.488	0.815	3.82	59.9
C2p15 page 47	3.11	0.574(5) 1.459	0.192(3) 0.488	0.816		59.8
C2p216	3.25	0.573 1.455	0.202 0.513	0.853		59.8
C2p217 sur chipped	3.22 add 0.02 (3.24) calc	0.573 1.455	0.202 0.513	0.853		59.6+

The above understood and witnessed by _______ Date _______ and by _______ Date _______

 $SrTiO_3$ GB Doping IBM Technical Notebook

10g $SrTiO_3$ w/ 2 wt % $B_{12}O_3$ added

Sp. g - 8.8 m.p. 820°C

10g + 0.2g $B_{12}O_3$ ⇒ 10.2

0.2g A_2O 7.14 g/cc Decomposes above 300C

$AgNO_3$ ⇒ mp 212C ; bp ⇒ decomp 169.8749 mw

~~4.328~~ g/cC

0.2g Ag_2O x $\dfrac{\Rightarrow 231.7394}{169.8749\ g\ AgNO_3/M}$ → 0.733, 1.364

0.2g Ag_2O x $\dfrac{\cancel{169.8749g\ AgNO_3/cc}}{\cancel{231.7394\ g\ Ag_2O/cc}}$ = 0.1466 ≈ 0.15g OK

X2 = 0.29

0.2g $AgNO_3$ x $\dfrac{169.874g}{}$

$2AgNO_3 \xrightarrow{\Delta} Ag_2O + 2NO_2 + O$

~~0.2g Ag_2O x $\frac{231.7394g\ Ag_2O/M}{169.8749\ g\ AgNO_3/M}$ = 0.273g $AgNO_3$~~

3.20, 3.23 3.92

Date and sign every entry/Ha ver sibly important entry witnessed. Submit an Inv osure of anything possibly new and inve

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Fre 3/5

I275000 Doped $SrTiO_3$ pellets

$SrTiO_3$ basis

STA-3 deformed, save powder,

STA-1	3.10	0.581	0.196		3.64	75.7	
		1.476	0.498	0.852			
	3.0	0.525	0.178		4.75	98.8	
		1.334	0.452	0.632			
STA-2	3.25	0.581	0.208		3.65	75.9	
		1.476	0.521	0.891			
	3.14	0.525	0.185		4.78	99.4	← polish 1
		1.334	0.470	0.657			
I24 STB-1	3.03	0.587	0.191		3.58	74.4	
		1.491	0.485	0.847			
	2.88 ~~2.92~~	0.590	0.172		4.47	92.9	
(chip)		1.370	~~0.437~~ 0.437	0.64(46)			
STB-2	3.17	0.586	0.197		3.645	75.9	
		1.488	0.500	0.869(5)			
	3.03 ~~3.21~~		0.179		4.52	94	
		1.370	0.455	0.671			
I275000 STB-3	3.27	0.583	0.237		3.66	76.1	
		1.481	0.60	1.03			
	3.61 ~~"3.74"~~	0.534	~0.216		4.55	94.6	← polish 1
deformed		1.366	0.549	0.793			
ST-D1	3.62	0.585	~0.235 varies	1.03(5)	3.50	72.8	
		1.486	0.597				
	360 ~~"3.67"~~	0.527	0.210		4.77	99.2+	← polish 1
		1.339	0.533	0.7505			

Comments - green D's fairly consistent, even w/ pressure variation

The above understood and witnessed by ________ Date ________ and by ________ Date ________

37/22000

DD-X	2.88	0.573	0.169		4.04	63.4%
		1.455	0.429	0.713		
	2.83	0.508	0.144			93
		1.290	0.366	0.478		
DD-Y	2.99	0.575	0.174		4.04	63.4%
		1.460(5)	0.442	0.740(5)		
	2.93	0.509	0.149			92.4
		1.293	0.379	0.498		

10°C/MIN RAMP IN NEW Al_2O_3 CRUCIBLE ON FRESH DD powdr.
975C for 2 HOURS & QUENCH. 20 MIN O_2 purge.

HORIBA CAPA-500
PARTICLE ANALYZER

DATE Bi_2O_3
SAMPLE 2/24/
SOLVENT 150

• CONDITIONS

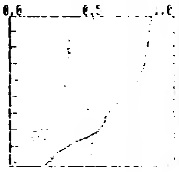
SOLV.VISC 2.79 (CP)
SOLV.DENS 0.79 (G/CC)
SAMP.DENS 6.80 (G/CC)
D(MAX) 10.0 (µm)
D(MIN) 1.00 (µm)
D(DIV) 1.00 (µm)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 0 SEC

• DATA

TIME ABSORBANCE
0.0 0.5 1.0



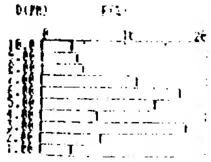
• DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
10.0 <	10.0	10.0
10.0- 9.0	3.5	13.5
9.00-8.00	4.2	17.7
8.00-7.00	4.8	22.5
7.00-6.00	11.6	34.1
6.00-5.00	16.8	50.9
5.00-4.00	13.8	64.7
4.00-3.00	6.8	71.5
3.00-2.00	17.6	89.1
2.00-1.00	7.2	96.3
1.00-0.00	3.7	100.0

D(AVE) 5.8 (µm)

• DISTRIBUTION GRAPH (BY VOL.)

D(µm) F(%)
0 10 20



HORIBA CAPA-500
PARTICLE ANALYZER

DATE Bi_2O_3
SAMPLE 2/24
SOLVENT 150

• CONDITIONS $\overline{V} = 0.69$

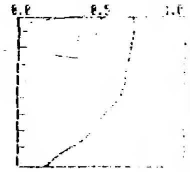
SOLV.VISC 2.79 (CP)
SOLV.DENS 0.79 (G/CC)
SAMP.DENS 8.00 (G/CC)
D(MAX) 10.0 (µm)
D(MIN) 1.00 (µm)
D(DIV) 1.00 (µm)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 0 SEC

• DATA

TIME ABSORBANCE
0.0 0.5 1.0



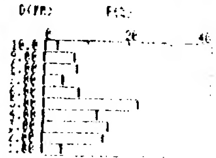
• DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
10.0 <	0.0	0.0
10.0- 9.0	2.9	2.9
9.00-8.00	7.1	10.0
8.00-7.00	7.6	17.[illegible]
7.00-6.00	4.4	22.[illegible]
6.00-5.00	8.3	30.[illegible]
5.00-4.00	22.4	52.8
4.00-3.00	12.8	65.[illegible]
3.00-2.00	15.5	81.[illegible]
2.00-1.00	14.5	95.[illegible]
1.00-0.00	4.5	100.0

D(AVE) 4.12 (µm)

• DISTRIBUTION GRAPH (BY VOL.)

D(µm) F(%)
0 20 40



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/24/88
SAMPLE C2-PIII, PASS III
SOLVENT 150
no Selective milling
clean machine

• CONDITIONS

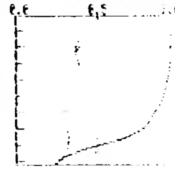
SOLV.VISC 2.10 (CP)
SOLV.DENS 0.79 (G/CC)
SAMP.DENS 6.30 (G/CC)
D(MAX) 10.0 (µm)
D(MIN) 1.00 (µm)
D(DIV) 1.00 (µm)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE
0.0 0.5 1.0



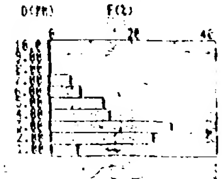
• DISTRIBUTION TABLE (BY VOL.)

D(µm)	F(%)	F(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	5.1	5.1
6.00-5.00	7.6	12.7
5.00-4.00	12.8	25.5
4.00-3.00	14.3	39.8
3.00-2.00	25.7	65.6
2.00-1.00	24.7	93.7
1.00-0.00	6.3	100.0

D(AVE) 2.65 (µm)

• DISTRIBUTION GRAPH (BY VOL.)

D(µm) F(%)
0 20 40



3/15 DC batch II $SrTiO_3$

jar tare 206.11 (206, unstable
256.06(?)
49.95 g loss 0.05 (0.1%) desired 50g

27.062
283.122 target
283.13 actual
27.07 +0.008
77.02 total

88.34(5) Pt crux tare
77.02 total above
165.36(?) expected comb. wght
165.34
.03 g error max. ✓✓ OK. (0.04% error)

~184.54 (19.20 tare) ✓✓ expect ~ 150.0 w/out top

150.15 after cooling!

3/16 yield < 100 mesh 59.85 g

3750/25,000

ST-D2	3.04	0.583	0.206		3.38	70.3 %
(2.9μm)		1.481	0.523	0.900		
M II						
	3.017	0.514	0.181		4.90	1.02 %
		1.306	0.460	0.616		

Date and sign every entry. Hav … ver … sibly important entry witnessed. Submit an Inv … osure of anything possibly new and inve…

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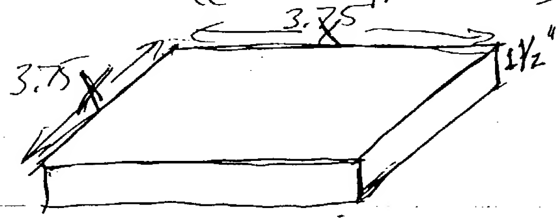
7070 GLASS count

$1\frac{1}{2}''$

7070 density – 2.13 g/cc $\Rightarrow \frac{kg}{1000g} \times \frac{lb}{3.73 \times 10^{-1} kg} = \cancel{0.00571 \frac{lbs}{cc}}$

$0.00571 \frac{lbs}{cc} \times \frac{16.387 cc}{in^3} = \cancel{0.0936 \frac{lbs}{in^3}}$ troy conv.



$4.08 \times 4.08 \times 1.5 = 21.32\ in^3$

$\cancel{21.32\ in^3}$ 24.9696 $\times 0.08 \frac{lbs}{in^3} = 1.997$ lbs

$1.5 X^2 \left(\cancel{0.0936 \frac{lbs}{in^3}}\right) = 2$

$1.5 (0.08\ lbs/in^3) X^2 = 2$ lbs 4.78

$0.1200 X^2 = 2$ lbs

$X^2 \simeq 16.66$

$X = \cancel{3.77}\ 4.08$

check density conversion : $\cancel{2.13 \frac{g}{cc} \times \frac{kg}{1000 g} \times \frac{lb}{3.73 \times 10^{-1} kg}} =$

OK $\quad \frac{0.00213 kg}{cc} \times \frac{1 lb}{0.435 kg} = 0.0048965 \frac{lb}{cc}$

1 lb = 4.535×10^{-1} kg
lb = 0.435 kg

$0.0048965 \frac{lb}{cc} \times \frac{16.387 cc}{in^3} = 0.08$

4 X 4 X 1.5 OR 5 x 5 x 1

@ 1" thick $\quad 0.08 X^2 = 2$
$X^2 = 25$
$X = 5$

The above understood and witnessed by ____ Date ____ and by ____ Date ____

3/21 1st pellet 700C for 12 hr ⇒ start @ 3 to ramp @ 10°/min) ... soak for 16-17 hrs.

C2P12 for green data on all pellets see pg 37

C2P13 2nd pellet 750C ... to run concurrently

Peter,

Since we didn't get to discuss this experiment in more detail, here is what needs to happen.

5 pellets — C2 overnight Init.
1st) - 700° C ~12 hr O_2 3.08
2nd) - 750° C ~12 hr O_2 3.02
3rd) - 800° C " "
4th) - 850° C " "

After the intermediate temperature anneal, weigh and measure each pellet. If no sintering, or at least a negligible amount, has occured, then refire each sample for 12 hrs again at the same intermediate temperature and then sinter each pellet for 2 hrs at 950°C. Ramp from the intermediate T to 950°C fast (~20°C/min).

} 965C used

Also sinter the 5th pellet at 950°C for 2 hrs, this is the control pellet. Thanks and have a good week.

Duane

~9:00 a.m. w/17 HRS

C2P12	3.06 (Δ-0.02)	0.572	0.191	no sintering, but 0.65% weight loss		
	3.01	0.50	0.163		5.65	88.7
		1.27	0.414	0.533		CRACKING = closing
C2P13	3.01 (3.005) (Δ-0.01)	0.572	0.187	no sintering, but 0.60% weight loss		
	2.97	0.504	0.163		5.57	87.4
		1.280	0.414	0.533		NO CRACKING "open"

3/23: Temp raised to 965 @ 20/min @ 9:00 → to temp @ 9:15 am

The above understood and witnessed by ____________ Date ______ and by ____________ Date ______

3/21 $SrTiO_3$ DRC-batch2 ⇒ fine cell after 3rd milling ⇒ 11g yield
+ 50g medium
61g / 72 orig.
~ 85% yield

after cleaning ~ 2g loss to machine
3g loss to blow-out

approx. expectations { 18% fines
82% medium

fines ~> 1.34 μm ave d̄
medium III → ≳ 2.2 μm range (2.2-2.8)

STD~~X~~1f-1 0.570 0.~~2~~12 3.09 64.2% {versus 72 on 3μm powders
2.74 1.448 0.538(5) 0.887

In furnace ~ 3:00 p.m., tripped off @ 975 2X, cooled to 1400C, then to 1600C.
Temp recovered/reset to 1650 @ ~4:30 p.m..

3/22 Ran all nite! 24 hrs @ 3:00 p.m. tuesday, 42 hrs. @ 9:00 AM Wends.
2.70 0.487 0.178 4.97 1.033% same as old's
1.237 0.452 0.543

Peter:

✓ started • Cut, section and polish, Cu-Bi [illegible] (Short Plan) start/finish 21/22

• Try firing one pellet of $SrTiO_3$ @ 1350°C overnight

• Try slip casting a pellet of $SrTiO_3$

→ fines Jet mill $SrTiO_3$ powder down to ~1μm. This will require "fixing" the jet mill. Then fire 1650°C overnight. USED fines, fire 21/22

✓ • Make another batch of $SrTiO_3$?

△

tare 202.33 *approx. due to instability - seems close: sometimes stable
50.00
252.33 target
act wght 252.33 *
Δ 0.0
27.062
279.392
act weight 279.4
~Δ 0.01

TRANSfer. 165.22 act wght
tare 88.17
77.05 wght mix
77.07 expected
~ 0.03% loss

POST/16HR 150.02
+ 14.87 g ~ expected loss
164.89
15.2 g actual loss

write-up 3/23, powder 3/24 MORN

3/22

C3-Synthesis (Reference)

Synthesis

$BaCO_3$
tare → 277.72 to zero
weight: ?
3/23 398.24-6 total
120.52-54 (−0.02%)

CuO
tare → 0.89 → zeroed
weight: 72.58 (7/9) 3/23
transferred quant

Y_2O_3 tare 0.85 → z'd 3/23
weight 34.35
transferred quant, +0.03%
total expected → 227.46

14 C2 Batch → $Y_1Ba_2Cu_3O_7$ 200g
from C1 batch calc. (pg 54 Book III → 72 Book II)

	at. Frac.	Redy	Mult.	final
Y_2O_3 →	17.1535 →	17.1907	→ x2	34.34
BaO_x →	46.5934	100	→ x2	93.1868

$BaCO_3$ conversion: 93.1868 $\frac{197.35}{153.34}$ = 119.932(3) ÷ 0.99 → 121.14(4)

CuO → 36.25(5) → 36.2893(4) → x2 72.57(9)

O.K. everything is Ba rich by analysis, so simply not ~~not~~ correct → 119.93
Apply ½. .606 120.54

$BaCO_3$
tare: 279.67
+ 120.54
400.21 won't read, but will tare

reads: 120.57(4-6) ooo 4/5

CuO
tare: 0.86/7
reads 72.58 transferred quant. tare to zero of paper

Y_2O_3 transferred quant
34.34/5 paper weighs ~0.1 when checked due to static from glove charge, but after glove/charge removal 0.00. Think OK when ... glove was (not more than 0.3% error)

Expected total weight} 227.46 g dry

BaO - 5.72 g/cc $BaCO_3$ - 4.43 Y_2O_3 - 5.01 CuO - 6.5-6.49
∴ If bumping occurs or selective loss, $BaCO_3$ should preferentially be lost ~~if~~ not ~~...~~ uniformly suspended.

Overnite @ 70C in 30" vacuum after "bump-free" isopropanol MIXING

3/24

	#1 cruc		#2		
	230.56		230.41		
	116.57		117.17		
	113.99	+	113.24	→ 227.23	0.1% loss or 99.9% Recovery

Prior to removing from bkr after overnite, cake broken up and "pulverized", then let cool under vacuum to remove any solv. resid.

In furnace @ 12:30 500C/HR RAMPS, 955 RXN T in flowing oxygen.

3/25 POST

			total	
219.22	218.34			
102.65	101.17	→	203.82	
101.79	100.34		202.13	0.83% loss
218.36				

318.71

Date and sign every entry. Have [illegible]sibly important entry witnessed. Submit an Inve[illegible]osure of anything possibly new and inventive.

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3/23 from pg 44 TREATMENT INQUIRY

C2P14 & C2P15 ⇒ original green & info on pg 37; both 3.11 then & now

C2P14: (800°C pretreat), purge - 2:20 p.m. 59.9 r.d. : 18 HRS
3.09 0.573 0.191 3.83 60.1 no appreciable sintering
1.455 0.485 0.806
3.07 0.522 0.171 5.125 80.5 apparently sintering appreciable
1.326 0.434 0.599

C2P15: (850°C pretreat) purge as above - 59.8 r.d. : 18 HRS
3.09 0.565 0.187 4.02 63.1 slight amount of sintering
1.435 0.475 0.768
3.08 0.531 0.179 4.87 76.5% initial sintering "appreciable"
1.319 0.442 0.632

C2P18 (CONTROL) 37/27500
2.92 0.574 0.180 3.83 60.1% O.K.
1.458 0.457 0.763
2.86 0.501 0.152 5.82 91.4
1.273 0.386 0.491

3/28
Post 48 hr. (2nd 24) - C3 batch

pre → 318.71
post → 316.88
(-)1.83
202.13
200.30
199.23 initial grd. yield

 3/23 from page 45 **IBM Technical Notebook**

STD-1f grain size slightly larger – interior fairly uniform ~25 μm ave w/ occasional 50 μm grain

~~Re-fired~~ Re-sintered overnite to check for additional growth.
Further polishing of 40 hr sample slice yields numerous 40-50 μm GRAINS! Growth seems probable!

3/24
Summary from 45

Green	2.71	1.448	0.538(5)	0.887	3.09	64.2
42 HRS. @ 1645C	2.70	1.237	0.452	0.543	4.97	100.33

slice back up to 1650 @ 5:00 PM (1 hr earlier due to control couple failure)
63 HRS SHUT off @ 1:45 PM 3/24 (21 hr)

3/24 from pg 45

after additional 12 hr RXN time 150.02 − 149.81 = 0.21 g : Assume constant now

total loss:

165.22	initial			
150.02	16 HR	− 15.20	98.6 %	Reacted
149.81	28 HR	− .21	1.4 %	cause for minor porosity?
		15.41		

> 61 gs recovered after mortar grinding.

> 2HRS on shaker mill w/ 5mm balls. — PSD 2.91 μm ave. flatter than jet, but not much better ø size,

> 60.4 g shaker yield

> 48.8 g MI JET YIELD

Slip-cast calculations: die 0.9" i.d. ⇒ 2.286 cm 0.3" (0.762 cm) desired green thickness

$\frac{\pi \cdot (2.286)^2}{4}(0.762) = 3.1275\ cc\ (4.81\ g/cc) = 15\ g\ SrTiO_3\ (0.6) = 9.0\ g$

(0.6) approx density

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Date and ... very entry. Have every possibly important ... entr ... Submit an Invention Disclosure of ... any ... possibly new and inventive.

IBM Technical Notebook

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/21/88
SAMPLE SrTiO3-DRC2
SOLVENT IS0
MIII

• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

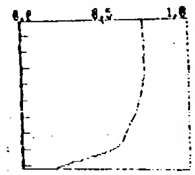
SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE

0.0 0.5 1.0



• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.0	1.0
9.00-8.00	0.0	1.0
8.00-7.00	1.0	2.0
7.00-6.00	2.9	5.[illegible]
6.00-5.00	9.5	15.[illegible]
5.00-4.00	12.5	27.[illegible]
4.00-3.00	12.1	39.[illegible]
3.00-2.00	12.6	52.[illegible]
2.00-1.00	39.6	[illegible]
1.00-0.00	7.9	100.0

D(AVE) 2.20 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 20 40

HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE SrTiO3
SOLVENT
MII

• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

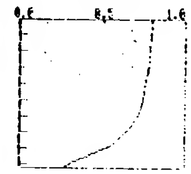
SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE

0.0 0.5 1.0



• DISTRIBUTION TABLE (BY VOL.)

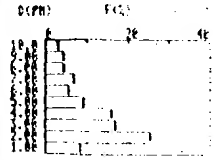
D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	3.1	3.1
9.00-8.00	4.2	7.3
8.00-7.00	4.5	11.9
7.00-6.00	6.9	18.8
6.00-5.00	5.4	24.2
5.00-4.00	9.2	33.4
4.00-3.00	16.6	4[illegible].4
3.00-2.00	17.0	66.[illegible]
2.00-1.00	25.2	91.6
1.00-0.00	8.4	100.0

D(AVE) 2.96 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 20 40



DPS

HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE SiTiO3
SOLVENT
MII

• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

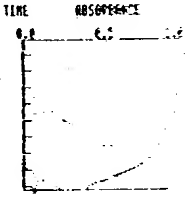
SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE

0.0 0.5 1.0



• DISTRIBUTION TABLE (BY VOL.)

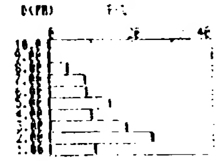
D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.[illegible]	0.[illegible]
8.00-7.00	4.[illegible]	4.5
7.00-6.00	8.4	12.9
6.00-5.00	8.7	21.6
5.00-4.00	14.4	36.[illegible]
4.00-3.00	5.5	4[illegible].[illegible]
3.00-2.00	18.4	64.[illegible]
2.00-1.00	24.5	83.[illegible]
1.00-0.00	16.7	100.0

D(AVE) 2.7[illegible] (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 20 40



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/21/88
SAMPLE SrTiO3-DRC2
SOLVENT IS0
MIII-5

• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

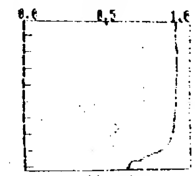
SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE

0.0 0.5 1.0



• DISTRIBUTION TABLE (BY VOL.)

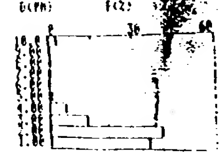
D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	2.9	2.9
9.00-8.00	0.0	2.9
8.00-7.00	0.0	2.9
7.00-6.00	0.0	2.9
6.00-5.00	0.0	2.9
5.00-4.00	0.0	3.0
4.00-3.00	5.9	9.6
3.00-2.00	13.4	23.0
2.00-1.00	41.0	64.[illegible]
1.00-0.00	35.9	100.0

D(AVE) 1.34 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 30 60



The above understood and witnessed by: Date and Date

Date and sign every entry. Have every entry witnessed. Submit an Inver ibly important anything possibly new and invent sure of

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3/29

10g $SrCO_3$ ⇒ 0.06774 moles
5.412g TiO_2

15.412g total

15.08 after mixing RECOVERY
.33

4/25/93 Note
Density calculations here were done using ρ $SrTiO_3$ lit. which is now known to be in error. It is 5.116 not 4.82

Dave's unreacted $SrCO_3/TiO_2$ } new batch $SrTiO_3$ ⊛ -

2.975	0.584	0.242				
2.42	0.458	0.190		4.745	98.6 !	NOT TOO GOOD
	1.16	0.483	0.510			
⊛ 2.91	0.585	0.194				
2.90	0.438	0.173		4.785	99.5 !	NOT TOO GOOD
	1.326	0.439	0.606			

The above understood and witnessed by ____ Date ____ and by ____ Date ____

52 A/1 $SrTiO_3$ grain growth IBM Technical Notebook 1470C

pellet 2 of fines

2.22	0.572	0.169		3.12	64.9%	~same as before
	1.453	0.429	0.711			
2.19	0.487	0.144		4.98	~~100~~ 103.5%	= 103.5
	1.237	0.366	0.440			
2.71	0.580	0.176		3.56	74.0%	
Duane's xcess	1.473	0.447	0.762			
TiO_2 estimate on 1/2	0.518	(0.262) 0.160		4.86	→ 1.01	101.–
est. 35	1.32	0.406	0.556			
(2.70)						

~ O.K. by wght?

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IBM Technical Notebook

$SrTiO_3$ doping

				M.W.		~~M.P.~~ 53
	0.0676	X2 0.135 ~~25~~ 25g batches		79.90		~~0.0676~~
$SrCO_3$	0.2 excess ~~0.125~~ ~~0.125~~	0.25		147.63		
Al_2O_3	STOIC 0.086	→ SAME	as nitrate	101.96		
V_2O_5	0.1754	0.308		181.88	3.357	690C
$SrTiO_3$				183.5182		

		moles	
13.531	TiO_2	0.16935	0.00084675 = ½ mole %
25.00	$SrCO_3$	↓	
38.531			

0.00084675 moles (79.90) → -0.06766 + 13.531 = 13.46

0.00084675 (147.63) → -0.125 + 25. = 24.875

Summary of additions, quantities

		excess	SUB 1	SUB 2	excess 2
	TiO_2 Ca	13.5986 13.6662	13.445 ✓*	13.531 ✓*	13.531
X2	$SrCO_3$	25.00 ↓	25.00 ↓	24.846	25.125
X2	Al_2O_3	—	0.086*	—	—
	V_2O_5	—	—	0.154* 0.16	—

* these quants are X2 since there are 2 moles of Al & V in Al_2O_3 & V_2O_5

Correction: $\frac{.16}{147.63}$ = 0.00108 moles $SrCO_3$

$\frac{0.00108 \text{ moles}}{0.00169}$ = 64% with loss due to decanting approx 70% stoic or 30% off addition excess V_2O_5

The above understood and witnessed by ______ Date ______ and by ______ Date ______

Sub2 V_2O_5 1 mol %, stoichiometric / not excess (see pg 53) :

25.0 g $SrCO_3$ weighed & transferred to beaker
0.16 g removed
0.16 g V_2O_5 added (~20cc saturated in hot water, decanted)

MISTAKE, now uncorrectable. Should have been:

0.00084675 (2) = 0.0016935 g $SrCO_3$ removed ∴

0.0016935 (147.63) = 0.25 g however, decanting over residual powder reduced actual V_2O_5 addition, and though nonstoichiometric (slightly) will use to see what happens.

38.02 yield after overnite vac @ ~90C
38.53 initial
0.51 loss in mixing 1.3 %

88.88 tare (zeroed)
38.08 .06 extra due to final beaker scrape ✓

In furnace to temp by 12:00, 4/6/88 16 HRS 8 A.M 4/7

'Severe' sintering, dark black appearance of powder body.

126.96
117.68
+ ~.05 g spillage
117.73

25g $SrCO_3$ × $\frac{\sim 103.62}{147.63}$ ≅ 17.55

126.96
− 117.68
9.28 ! some bound water?

~7.40 g expected loss

26.18 g ground yield

See 57 & 58 for sintered …

The above understood and witnessed by ______ Date

and by ______ Date

Sub 1 Al_2O_3 → 1 mol% added as nitrate

0.00084675(2) = 0.0016935 moles // $Al(NO_3)_3 \cdot 9H_2O$ 375.14

½ mol% 1 mole % Al 1:1 so use 0.0016935 moles

0.0016935 moles Al nitrate $\left(\frac{375.14\,g}{mole}\right)$ = 0.6353 g

(303172)

So remove 0.0016935 moles TiO_2 ∴ 0.0016935(79.9) = 0.135 g TiO_2

13.531
− ~~0.135~~
13.396 g TiO_2, 0.6353 g Al nitrate in soln

38.29 mix yield
39.03 theoretical
0.74 mix loss 1.9%

39.03
− 0.46
38.57
38.27 ⇒ 0.3^+ mix loss

0.6353 g $\left(\frac{101.9612}{375.14}\right)$ = 0.173 − ΔOA6

89.55 tare (fused)
38.27 NOTE: nitrate decomposes in hot water. Must explain some of loss.

In furnace to temp by 12:00 p.m., 4/6/88 ⇒ 16 hrs 8 AM 4/7

Little sintering of powder, light $SrTiO_3$ color, mottled.

118.54
127.82
− 9.28 g ! SAME AS SUB 2! ~~had~~ giving the same, even though exact loss is coincidence.

Yield ⇒ 27 g

See 57 & 58 for SINTERED DATA

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Date an... sig ...ry entry. Have every possibly important entry ... ...submit an Invention Disclosure of anythin... ...sibly new and inventive.

Phase DIAGRAMS (arrows) for SrO·TiO2 system, and assorted others

SrO–SiO$_2$

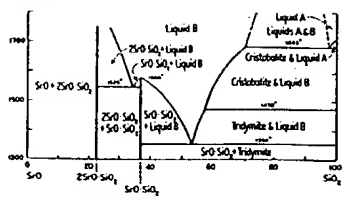


FIG. 296.—System SrO–SiO$_2$.

P. Eskola, *Am. J. Sci.*, 5th Ser., 4, 336 (1922); modified by J. W. Greig, *ibid.*, 5th Ser., 13, 19 (1927); see also F. C. Kracek, *J. Am. Chem. Soc.*, 52 [4] 1440 (1930).

ZnO–Al$_2$O$_3$

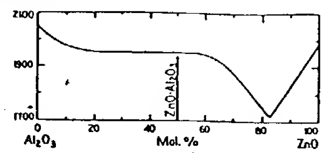


FIG. 299.—Liquidus curve of system ZnO–Al$_2$O$_3$.

E. N. Bunting, *Bur. Standards J. Research*, 8 [2] 280 (1932); R. P. 413.

ZnO–B$_2$O$_3$

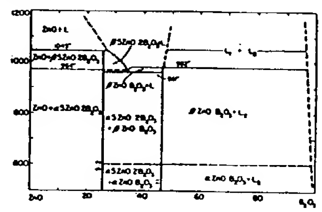


FIG. 300.—System ZnO–B$_2$O$_3$.

D. E. Harrison and F. A. Hummel, *J. Electrochem. Soc.*, 103 [9] 496 (1956); see also, "Structure of Zinc Metaborate, Zn$_4$O(BO$_2$)$_6$," P. Smith, S. Garcia-Blanco, and L. Rivoir, *Anales Real Soc. Espan. Fis. Quim.* (Madrid) *Ser. A* (Nov.-Dec.), 263–268 (1961).

SrO–TiO$_2$

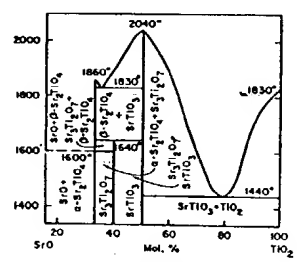


FIG. 297.—System SrO–TiO$_2$.

Miroslawa Dryś and Wlodzimierz Trzebiatowski, *Roczniki Chem.*, 31, 492 (1957).

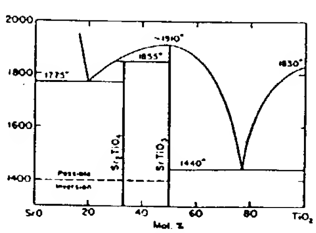


FIG. 298.—System SrO–TiO$_2$; tentative.

Rustum Roy; private communication, 1957.

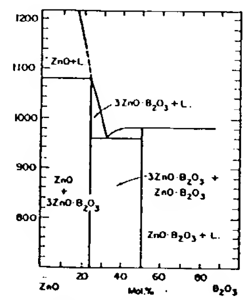


FIG. 301.—System ZnO–B$_2$O$_3$.

Yu. S. Leonov, *Zhur. Neorg. Khim.*, 3, 1246 (1958).

SrO–TiO$_2$

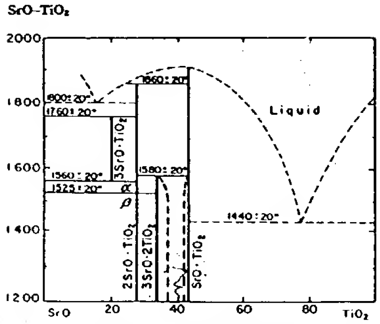


FIG. 2334.—System SrO–TiO$_2$. 3SrO·2TiO$_2$ ss extends to approximately the 4SrO·3TiO$_2$ composition.

Antonio Cocco and Franco Massazza, *Ann. Chim. (Rome)*, 53, 892 (1963).

SrO–ZrO$_2$

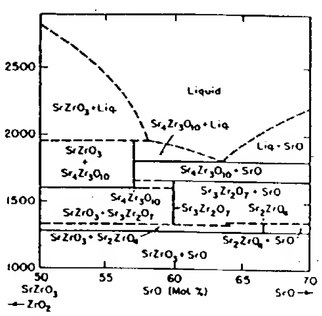


FIG. 2335.—System SrO–SrZrO$_3$.

Gilbert Tilloca and Monique Perez y Jorba, *Rev. Hautes Temp. Refractaires*, 1 [4] 337 (1964).

The above understood Date and Date

Excess $SrTiO_3$ (Excess 2) (Excess 1)

13.531 TiO_2 → 13.53 — 25.25 $SrCO_3$ 25.28 | 13.6 TiO_2 13.61 25.00 Sr 25.02 .03 t loss

zero off after addition probably static

In drying oven @ ~100C under house vacuum @ 1:30 p.m. 4/6/88

38.72 after drying
87.87 crucible
126.59 w/ addition
118.85
7.74

38.43 after drying
87.07
125.50
117.79
7.71

$\frac{103}{147}$

$25g \times \frac{101.962}{375.14} = \cancel{6.795}\ 17.5$ g

$\cancel{6.795/7.7 = 88.25\%}$ 12% excess loss
7.5 / 7.7 ≙ 97.5% +

#1 ← TiO_2 | #2 ← $SrCO_3$

NO GREEN DATA TAKEN → NO GREEN DATA

2.75	0.520	0.159		4.96	1.03	3.01	0.517	0.175		5.0	1.04
	1.321	0.404	0.554				1.313	0.444(5)	0.60		
N_2					EXCHANGE						
2.71	0.529	0.160		4.71	97.9	3.00	0.520	0.176		4.89	1.02
	1.344	0.406	0.576				1.321	0.447	0.613		
SUB2						SUB1					
2.38	0.540	0.135		4.69	97.5	2.56	0.529	0.150		4.74	98.5
	1.372	0.343	0.507				1.344	0.381	0.5405		
N_2											
2.41	0.554	0.141		4.31	89.6	2.60	0.526	0.151		4.83	1.00
	1.410	0.358	0.559				1.336	0.384	0.538		

SUBSTITUTION 1 : Al_2O_3 1 mol % 16 HRS RXN.
" 2 : V_2O_5 ↓

	N_2	O_2
1 :	100.4	98.5
2 :	89.6	97.5

EXCESS DOPING 1 : ½ mol % TiO_2
" " 2 : ↓ $SrCO_3$

1 :	N_2	O_2
1 :	97.4	103
2 :	102	109

"MECHANICAL MANIPULATION"

fines : 103.4

fines ⅓, med ⅔ mix : 102.3

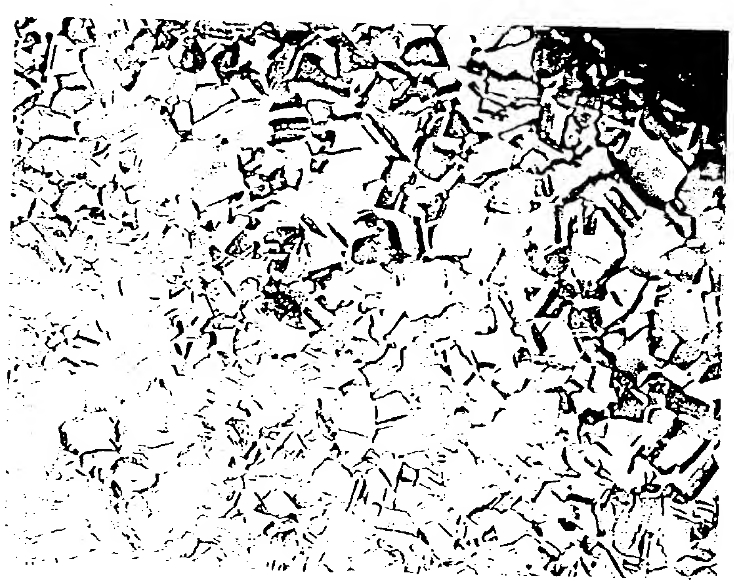
MED : 100.4

4/11 Pharr Cu/Bi studies

600C overnite N_2 treatment on as rcv'd material (Cu)

INTERIOR
INHOMOGENOUS
(ABNOMALOUS)
GRAIN GROWTH

100X



EXTERIOR
~HOMOGENEOUS

1, 3, 10% Bi in copper.

5 g total per batch

4.95 post 750C

g Bi : 5(0.01) = 0.05 5(0.03) = 0.15 ~~10~~5(0.10) = 0.5 g

Cu 4.95 4.85 4.50

actual 0.05-6 0.15-0.16 0.50-1

4.95-6 5.00 ~~4~~.99-5.0

recovery 4.87 added to 5.00 4.99 — 4.95

5.00

4.98 after 400C overnite (not disturbed) 4.90 after 400C overnite

Reloaded 1% & will continue w/ 3%. Will make a new 10% and a 50% and fire @ 750C. Crucible shortage ⇒ will likely modify above.

50/50 10% Bi 3% as above

2.5g Bi, 2.5 g Cu

g Bi : 2.51 actual 0.51

5.02(3) total

post Cu 5.01 4.51(2)

Cu 2.50

Post 750C Post 750C

cruc 1.34 1.32(3)

total 6.34 6.3 6.24 6.21

5.00 4.91 ~~5.01~~ ? 4.89

4.92 400C RESULT

NOTE: INITIAL 400C (420C) for low (1-10%) percentages of Bi did not produce expected densification/solidification of pucks.

D5 ; 35% Bi/Cu melts & 1 Bi crucible filling

25% 35

0.25(5) = 1.25 g Bi, 3.75 Cu 1.75 Bi ; 3.25 Cu

6.33 PRE 6.25(6) loaded
1.33 1.30(1)
5.00 4.95 starting total

6.30 POST Δ = 0.03 Bi 6.23 Δ = −0.02

Possible post density. 0.315, 0.385 8.28 90.
4.97 0.800, 0.978 0.6 definitely smaller volume, higher density

A/H2 ARGON/H2 Bi filling 13.32 POST

~~13.34~~ 13.34 PRE 25%
~~13.18~~ 1.26 Bi
filled 12.82 CRUX ⇒ 11.86 5.03 w/Cu
cruc: 11.39(40) 01.48
~~11.43~~
13.30 after slag removal

POST 12.81

ARGON/H2 25% Bi RUN ⟸ Pellet RUN

pellet: 4.87 0.485 0.222 7.25 79%
1.232 0.564 0.672

8.96 × 0.75 + 0.25 (9.8) =
~~9.8~~
6.72 + 2.45 = 9.17

Furnace started ... 2 HR purge ; 3:35"
4.84 0.483 0.234 ?? pellet bloating due to Bi vaporization?"

Bi_2O_3: sp. g. 8.8 m.p. 820°C

$SrTiO_3$ GRAIN GROWTH EXPERIMENT - MECHANICAL MEASURES

1) PSD weighting
2) fines full density & free sintering of polished surface
3) reacted to constant weight (last batch) sintering as in #2

FINES = F

2.04	0.570	0.155		3.14	65.3
	1.498	0.394	0.649		
2.01	~~0.488~~	0.132		4.975	10~~3.4~~
	1.239(5)	0.335	0.409		

fine/medium = FM

2.19	0.577	0.154		3.32	69.0
	1.465	0.391	0.659		
2.17	0.504	0.135		4.92	102.3
	1.28	0.343	0.441		

chipped M

2.46(7)	0.582	0.165		3.43(5)	71.4
	1.478	0.419	0.719		
2.46*	0.519	0.147		4.85	100.4
	1.318	0.373	0.509		

REMARKS → * some powder adhered

4-12-88

Sintering Regime

Rapid Temp w/ 10 cc/min O_2 4:25 p.m.

1550C initial set, after reaching temp for 1 HR, 1640C overnite

5:20 p.m. ~ 1100C, Tcontrol blown. ∴ ~30-45 minutes @ 1540.

Restarted @ ~5:55 & brought directly to 1640C.

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64 4-26-88

LEAK TESTING - S.C. TORR HIGH VAC

25 millitor after continued pumping through system
STARTING WITH Roughing valve

Will check pumpdown through HVAC valve alone tomorrow.

4/27 → VACATION
4/28 Pump down through high vac initially unsuccessful, must have been stuck valve, but after freeing can get down to ~50 millitor in 15 minutes. Will continue pumping.
1/2 40

Down to 10 thru Rough, 30 w/ HVAC only, Rather quick leak-back when both closed off indicating leaks in system:

1) FURNACE
2) Elbow connection
3) Pump

See page 78

5/13 Promised CSS test Monday (Chuck!)

The above understood Date and by Date

Date and sign every entry. Have every entry witnessed. Submit an Inver[redacted]D. anything possibly new and inven[redacted]ibly important ure of

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5/13

First Milling - AutoFeed - Teflon liner C-3

48.17 g known, but "few" occ before total processed wght. checked

49.69 g yield

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/13
AutoFeed SAMPLE C3-Pass I
SOLVENT ISO
Teflon liner

• CONDITIONS

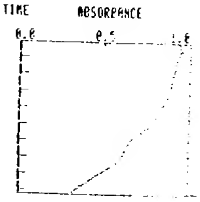
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

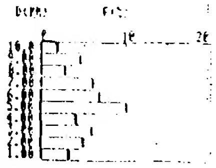


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	F(%)
10.0 <	49.7	49.7
10.0- 9.00	1.8	51.6
9.00-8.00	4.5	56.1
8.00-7.00	2.7	58.6
7.00-6.00	6.1	64.9
6.00-5.00	6.9	71.7
5.00-4.00	10.2	81.9
4.00-3.00	4.0	85.9
3.00-2.00	6.0	91.9
2.00-1.00	5.0	96.8
1.00-0.00	3.2	100.0

D(AVE) 9.65 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



PASS II 5/16

47.6 g yield : much fluffier, looks ~ like 5 μm pwdr.

IMMEDIATELY REMILLING

pg. 66 for PSD sheets ~2g loss!

PASS III 45.8

45.8

PASS IV 44.5

~18.0 5 pellets

26.5

← High large particle % 49.7 vs 18 for C2

← overall ave. ~10 vs 5-6 for C2

however distribution seems similar & some bypassing must have occurred.

Will work on Tuesday.

The above [redacted] and witnessed by ________________ Date ________ and by ________________ Date ________

Bi/Cu 25/75 crucible packed, sinter overnite in $Ar/H_2(5)$ @ 750C.

#1 ⇒ < ~~100~~ mesh Cu, spherical ("new")

#2 ⇒ ~~100 mesh~~ 10μm ~~spherical Cu~~ (old")

#3 ⇒ penetration 10μm

spherical

#2:	1.25/5.01	#1.	1.25/5.00
	6.34		6.35
cerx	1.34		1.35
	5.00		5.00
POST	6.31		6.32
	8.0–8.5mm ℓ		8.37
	9.65–9.75mm dia		9.65–10.0

After interruptions: 5/24 start cut & polishing

6/8/88 Finish: 6/8/88 after

{Porosity is reduced, and 3rd ∅ 'oxide' (grey) has been eliminated in forming gas.}

C3-P1-5 Green Data { Sinter → { Picked up mill teflon Material ruined

150275	G	mm	mm			
C3P1	3.59	1.470	~~0.79~~	0.80		70.5
	3.33					
		cm	cm			
C3P2	3.33	1.477	0.442	0.757	4.90	69%
	2.93					
C3P3	3.58	1.472	0.478	0.813	4.40	69%
C3P4	3.37	1.476	0.440			
C3P5	3.53	1.478	0.471		5.53	87 even w/ cracking puck OK. I think Mill no good.
	3.19!	1.331	0.415	0.577		

NOTES: PELLETS SEEM TO HAVE SHED ORGANIC/GAS?!
Looks like the pellet milled in plastic container.

15026 UNI 6700 20°/→550, 10°/→975, 2HRS,

C3P6	2.52	1.50	0.32		4.46	70.1%
Coarse				0.565		
	2.50	1.482	0.316	0.55	4.55	71%

Calcinations - 750C IN @ 8:00 P.M. 5/17/88 out 9:00 AM 5/18

TiO_2 - Cerac 39?

16.3620
− .8610
15.501 g TiO_2 weighed

89.4610 crux
105.0174 crux + TiO_2
15.5564 g TiO_2 by difference ~~99.64%~~ → 0.3% Δ + 0.0554

5/18
POST
105.0174
104.9395 GAINING ΔT > $ΔT_{CO_3}$
0.0779 / 15.501 = 0.5% +

EQ → 105.0884
105.0174
0.0090 / 0.0779 = 88.5% back

$SrCO_3$

18.4193
0.8720
17.5473 → 0.8752 / 17.5441 Δ 0.0032 ≈ ΔΔDif !!

109.9615
92.3660
17.5955 Δ + 0.0514 ΔΔDif ⇒ 0.004g ~ 4mg calibration

POST 109.9615
109.8870
0.0745 / 17.5441 = 0.4% GAINING → EQ 109.9510
− 109.9615
0.0105 85.6% back

5/19 TiO_2 { ~~$SrCO_3$~~ 2nd Cal POST $SrCO_3$

105.0174 −
104.8670 (−0.0003 cal)
.15
~1%

109.9615
109.8580 (−0.0003 cal)
0.1035
~0.6%

Date and sign every entry. Have every pos[illegible] important entry witnessed. Submit an Invention Discl[illegible] of anything possibly new and inventive.

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*Register with local Recorder

HF Silicon Etch/Wash/Buffer Solns.

{80g NH_4F in 120g H_2O (Distilled) → ~120cc 40 wt.% NH_4F REAGENT

~170cc

actual soln is 40:1 ~~BHF~~

BHF → 40 parts NH_4F reagent : 1 part HF (49 wt%) soln.

QUENCH → 10:1 DI : NH_4OH reagent 50ml : 500ml

BHF clean → 10:1:2.2 (NH_4F reagent : HF 49 : Glycerin)

16(10) = 160 16(1) = 16 16(2.2) = 35.2 = 211 ml

320

350

190 35 77

MSG:FROM: SARDESAI--FSHVMCC TO: MDT --YKTVMT 05/18/88 12:39:40
To: MDT --YKTVMT

From: Viraj Sardesai
8-533-8545, SCL Pers Metals,GTD E.Fishkill
IBM INTERNAL USE ONLY (Unless otherwise specified)
SUBJECT: BHF concentrations used in SCL

Michael,

We use 40:1 BHF for pre platinum, emitter screen ox removal and for s metal preclean.

The chemical is commercially available premixed solution and has 40 parts (by volume) of 40 wt pct NH4F solution mixed with 1 part of 49 wt pct HF solution. Both NH4F and HF are in aqueous solutions. Manufacturer specs the HF concentration to 0.61 to 0.77 moles per liter and specific gravity of 1.106 . } BHF

For S postL/O BHF clean 10:1:2.2 (NH4F:HF:Glycerin) is used prepeared similarly ,and quenched in 10:1 NH4OH solution (28 Wt pct NH4OH solution diluted to 10 times its volume in DI water). QUENCH BHF CLEAN

cc: SZECSY --FSHVMCC HOUGHTON--FSHVMCC

Regards,
VIRAJ
FSHVMCC(SARDESAI),D/11G B/322 Z/5T1
************* OUR TEAMWORK MAKES THE DIFFERENCE ! *************
--BHF concentrations used in SCL

The above understood and witnessed by ______________ Date ______

and by ______________ Date ______

Date and sign every entry. Have every poss… entry witnessed. Submit an Invention D… anything possibly new and inventive. important of

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*Register with local Recorder

5/18 Polymilling — mill #2

PASS 40g batch	YIELD	PSD 1	2	Δ
I	36.8	6.94	4.84	3
II	33.4	3.65	3.21	.3
III	31.3	3.53		2 bag changes required
IV	28.4	2.98 !		3 actually higher erroneous 7% @ 710
V	25.75	3.1 !		2.5 New bag

DO

The above understood and witnessed by ______ Date ______ and by ______ Date ______

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…stered IBM Confidential*
Register with local Recorder

Date and s… …ery entry. Have every possibly important entry … Submit an Invention Disclosure of anyth… …ssibly new and inventive.

ISO 28,

						MARGINAL GREEN DENSITIES	
C3P6	3.29	1.474	0.471	0.80	4.11	64.6	
	3.23	1.343	0.416	0.59	5.48	86.2 !	
C3P7	3.19	1.476	0.455	0.779	4.00	64.5	
	3.13	1.343	0.404	0.57	5.49	86.3	
C3P8	3.21	1.473	0.462	0.787		64.1	TO KING-TU
	3.16	1.341	0.409	0.577	5.48	86.2	
C3P9	3.08	1.474	0.441	0.7525	4.09	64.3	
	3.02	1.336	0.391	0.548	5.51	86.6	

NOTES: PELLETS IN @ 4:55 with flowing O_2 (bottled, dessicated)
Heating started @ 5:15 @ 20°/min (97C @ start)
@ 265C cut back to 10°/min; to reach sinter T @ 6:35 p.m.
Sintering @ 975C for 2HRS. till 8:35 p.m.
Quench & remove.

The above understood and witnessed by ____________ Date ____________ and by ____________ Date ____________

995C sinter

C3P10	3.18	1.474	0.453		4.114	64.7
				0.773cc		
	3.11	1.338	0.399		5.54	87.2
				0.561		

NO (appreciable) Δ dia. ∅ !

T3 "try" → MILL #1

yield	PASS	Δ			Ave. part dia, <100mesh
40.35	0				
29.~	I	10	!	1-2g leaks	3.29
21.5	II	8	!	~1g leak	3.11
18.5	III	3	!	4.0⁺ g of ~2.0µm pwdr in mill neck	

22.4 total mix yield

C3P10 - 18

975C →

C3P10 ①	3.14	1.461	0.473		3.975	62.50
	✓3.08	1.306	0.400	0.79 / 0.536	5.75	90.4
C3P11 C ②	3.28	1.462	0.495			
	✓3.21	~~1.~~308	0.417	0.56	5.73	90.0
C3P12 ③	3.04	1.463	0.450			~~89.31~~ ← RECHECK TOMORROW
	2.97	~~1.307~~ ~~1.310~~ 1.306	0.388 / 0.385	~~0.52~~ 0.523 / 0.516	5.68 / 5.756	90.5
C3P14	3.21	1.464	0.475			
	✓3.14	1.308 / 1.306	0.405 ✓	0.548	5.77-8	90.7-9

1000C →

C3P15	3.04	1.461	0.452			
	2.97	1.298	0.386	0.511	5.81	91.35
C3P16 (polished)	3.08	1.466	0.457			
	3.02	1.301	0.390	~~0.5~~ 0.519	5.82	91.5
C3P17	3.28	1.464	0.492		~~5.75~~ 5.82	90.4 + chip off
	3.22 (3.28)	1.298	0.422	0.548		91.5

IBM Technical Notebook

6/8/88

Centorr HVAC system close to finished with leak testing, preliminary operation checks.

System mechanical pump down < 20-30 minutes

Turbomolecular ↓ down → to 5×10^{-5} within 1 hr with attainments at mid 6's by 1:00 p.m. Start was 9:15 originally.

Block-off flange, new Centorr purge / plug fittings still needed. Foreline ~~leak~~ test reqd.

Bi/Cu Free-Crucible Sinter Vacuum Run

To glass shop 6/7/88. Batch size to be 4.0 g to allow for ease of manipulation during sealing of quartz tube.

4.0 (0.25) = 1.00 g Bi
↓ (0.75) = 3.00 g Cu (will use 10 μm Cu powder)

~~Bi → 1.00 (0.99)~~
~~Cu to 4.03~~
~~Cu 3.03~~
~~from JAR 4.02(1)~~
~~crux 1.33~~
~~w/ mix 5.33(4)~~
~~4.00(1)~~
~~4.01~~

~~~ 24.8 % ~ 25~~
~~75.2 % 75~~

Redo - spilled in GLASS SHOP

6/9 New crucible shape/size for stability
Lets take 5.0 g batch
1.25/5.0 (±0.01)

crucible 3.35
w/ mix 8.35
5.00

6/14 After overnite sinter & removal from quartz tube

cruc. & sinter 8.30 g (some spillage in tube before heat treatment)

no appreciable vapor product seen ~~on~~

Conclusions: Vacuum doesn't appear to work as well as Ar/H2. Sample full of holes, but no evidence of oxidation, so holes are real. Again, no evidence of vapor phase deposition in tube.
~~~

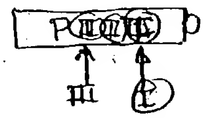
6/14/88

IBM Technical Notebook

To do: 10, 20, 25 % in Ar/H_2 (5) ~~4~~ MONDAY RUN! →(VACATION)

WENDS 6/22/88

5 gram batches: 0.25 (5) = 1.25 Bi / 3.75 Cu (I)
0.20 (5) = 1.00 Bi / 4.00 Cu (II)
0.10 (5) ~ .5 Bi / 4.5 Cu (III)



BOAT ORDER

(I) 5 gram w/ new conical crucible
mix 9.03
tare 4.03
5.00
↑
PRE

voids, but some areas seem OK. less small voids, some good regions versus (II), however large voids a mystery. Need repeating, maybe longer times.

(II)
9.00
tare 4.00
5.00

seems very good, no large voids, mic exam next, microscopic exam shows many small "pockets" or voids. Usually circular.

"old"

(III) 6.31
tare 1.31
5.00 'Nominal loss possible'

did not densify fully

The above understood and witnessed by ______ Date ______ and by ______ Date ______

Administrative Notes

SrTiO3 Milling Results

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/24/88
SAMPLE SrTiO3-B
SOLVENT 150
MI-MED

• CONDITIONS
SLIGHT IMPROVEMENT

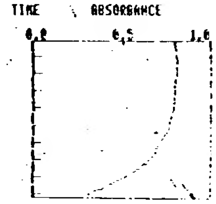
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA D= 0.8

TIME ABSORBANCE



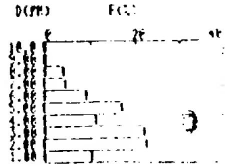
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	3.9	3.9
7.00-6.00	4.4	8.4
6.00-5.00	8.9	17.2
5.00-4.00	17.1	34.2
4.00-3.00	11.2	45.4
3.00-2.00	22.0	67.4
2.00-1.00	22.5	89.8
1.00-0.00	10.2	100.0

D(AVE) 2.79 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/21/88
SAMPLE SrTiO3-DRC2
SOLVENT 150
MIII-S

• CONDITIONS

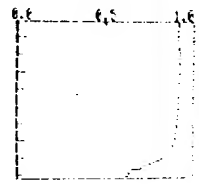
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE



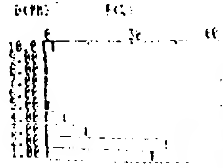
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	2.9	2.9
9.00-8.00	0.0	2.9
8.00-7.00	0.0	2.9
7.00-6.00	0.0	2.9
6.00-5.00	0.0	2.9
5.00-4.00	0.8	3.8
4.00-3.00	5.9	9.6
3.00-2.00	13.4	23.0
2.00-1.00	41.0	64.1
1.00-0.00	35.9	100.0

D(AVE) 1.34 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/22/88
SAMPLE SrTiO3-DRC2
SOLVENT 150
MIII

• CONDITIONS

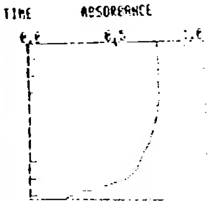
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE



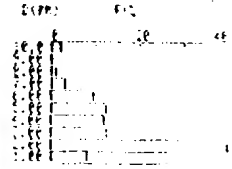
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.0	1.0
9.00-8.00	0.0	1.0
8.00-7.00	1.0	2.0
7.00-6.00	2.9	5.[illegible]
6.00-5.00	9.5	15.3
5.00-4.00	12.5	27.8
4.00-3.00	12.1	39.8
3.00-2.00	12.6	52.5
2.00-1.00	39.6	92.1
1.00-0.00	7.9	100.0

D(AVE) 2.20 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



Administrative Notes

C3 Milling Results – T1 ö Teflon T2 - Polyu T3 - PolyU "0"

T1

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PIV
SOLVENT 150

• CONDITIONS T1 →

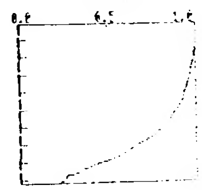
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE 0.0 0.5 1.0



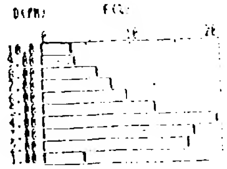
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	3.3	3.3
9.00-8.00	3.6	6.9
8.00-7.00	6.1	13.0
7.00-6.00	7.7	20.7
6.00-5.00	9.5	30.2
5.00-4.00	12.6	42.8
4.00-3.00	19.6	62.4
3.00-2.00	16.8	79.2
2.00-1.00	16.2	95.4
1.00-0.00	4.6	100.0

D(AVE) 3.63 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PIII
SOLVENT 15

• CONDITIONS D≈0.97

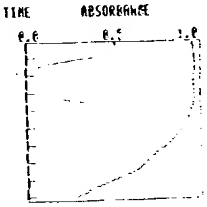
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE 0.0 0.5 1.0



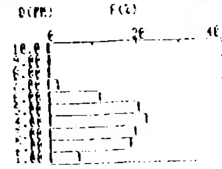
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	2.1	2.1
6.00-5.00	11.5	13.6
5.00-4.00	20.2	33.8
4.00-3.00	21.9	55.7
3.00-2.00	19.4	75.1
2.00-1.00	18.3	93.4
1.00-0.00	6.6	100.0

D(AVE) 3.26 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PII
SOLVENT 150

• CONDITIONS D~0.9

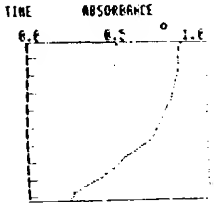
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE 0.0 0.5 1.0



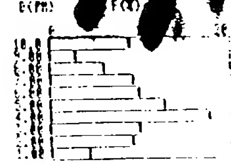
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	9.9	9.9
10.0- 9.0	0.9	10.8
9.00-8.00	2.8	21.6
8.00-7.00	6.0	27.7
7.00-6.00	9.2	36.8
6.00-5.00	9.8	46.6
5.00-4.00	12.6	59.2
4.00-3.00	17.6	76.9
3.00-2.00	9.8	86.7
2.00-1.00	9.0	95.8
1.00-0.00	4.2	100.0

D(AVE) 4.73 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



Administrative Notes

T2 Part I

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-PI-T2
SOLVENT 150

• CONDITIONS

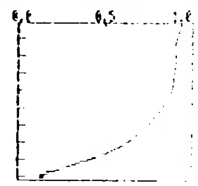
SOLV.VISC 2.10 (CP)
SOLV.DENS 0.79 (G/CC)
SAMP.DENS 6.30 (G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00 (µM)
D(DIV) 1.00 (µM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.9

TIME ABSORBANCE



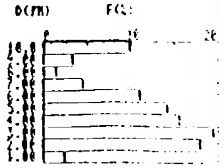
• DISTRIBUTION TABLE (BY VOL.)

D(µM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	9.8	9.8
9.00-8.00	3.4	13.2
8.00-7.00	1.5	14.8
7.00-6.00	4.5	19.2
6.00-5.00	11.2	30.4
5.00-4.00	14.1	44.5
4.00-3.00	15.6	60.1
3.00-2.00	19.4	79.5
2.00-1.00	18.0	97.5
1.00-0.00	2.5	100.0

D(AVE) 3.65 (µM)

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-PI-T2
SOLVENT 150

• CONDITIONS

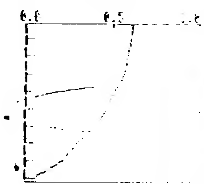
SOLV.VISC 2.10 (CP)
SOLV.DENS 0.79 (G/CC)
SAMP.DENS 6.30 (G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00 (µM)
D(DIV) 1.00 (µM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.57

TIME ABSORBANCE



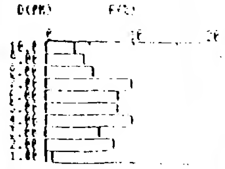
• DISTRIBUTION TABLE (BY VOL.)

D(µM)	F(%)	R(%)
10.0 <	30.4	30.4
10.0- 9.0	3.2	33.6
9.00-8.00	4.3	42.9
8.00-7.00	5.5	49.4
7.00-6.00	9.8	59.2
6.00-5.00	8.3	67.5
5.00-4.00	8.1	75.6
4.00-3.00	9.9	85.4
3.00-2.00	6.2	91.6
2.00-1.00	7.8	99.3
1.00-0.00	0.7	100.0

D(AVE) 6.94 (µM)

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-PI-T2
SOLVENT 150

• CONDITIONS

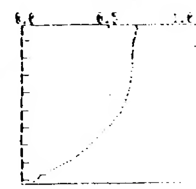
SOLV.VISC 2.10 (CP)
SOLV.DENS 0.79 (G/CC)
SAMP.DENS 6.30 (G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00 (µM)
D(DIV) 1.00 (µM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~.67

TIME ABSORBANCE



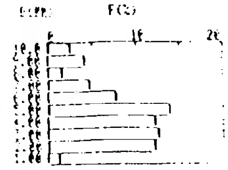
• DISTRIBUTION TABLE (BY VOL.)

D(µM)	F(%)	R(%)
10.0 <	27.1	27.1
10.0- 9.0	2.5	29.6
9.00-8.00	4.1	33.7
8.00-7.00	1.5	35.2
7.00-6.00	4.6	39.9
6.00-5.00	7.9	47.8
5.00-4.00	13.8	61.6
4.00-3.00	12.3	73.9
3.00-2.00	12.7	86.6
2.00-1.00	12.2	98.8
1.00-0.00	1.2	100.0

D(AVE) 4.84 (µM)

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



Administrative Notes

T2 PART 2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-P4-T2
SOLVENT 150

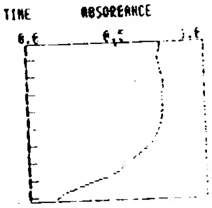
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.7

TIME ABSORBANCE



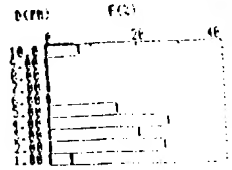
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	7.2	7.2
9.00-8.00	0.0	7.2
8.00-7.00	0.0	7.2
7.00-6.00	0.0	7.2
6.00-5.00	0.3	7.6
5.00-4.00	15.1	22.7
4.00-3.00	27.0	49.7
3.00-2.00	20.0	69.7
2.00-1.00	25.6	95.3
1.00-0.00	4.7	100.0

D(AVE) 2.50 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-P3-T2
SOLVENT 150

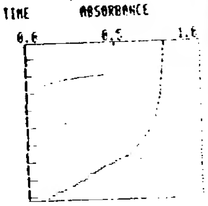
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.7

TIME ABSORBANCE



• DISTRIBUTION TABLE (BY VOL.)

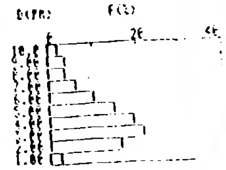
D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	2.1	2.1
9.00-8.00	3.9	6.0
8.00-7.00	3.7	9.8
7.00-6.00	6.2	16.0
6.00-5.00	10.1	26.6
5.00-4.00	15.0	41.6
4.00-3.00	19.6	66.1
3.00-2.00	21.4	81.5
2.00-1.00	16.0	97.5
1.00-0.00	2.5	100.0

D(AVE) 3.53 (PM)

NO MILLING / BAG CHANGE

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19/88
SAMPLE C3-P2-T2
SOLVENT 150

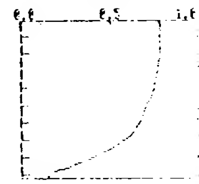
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE



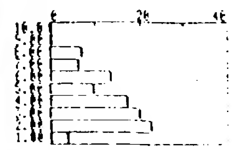
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	7.0	7.0
7.00-6.00	6.4	13.4
6.00-5.00	13.4	26.8
5.00-4.00	9.8	36.6
4.00-3.00	17.1	53.0
3.00-2.00	20.0	73.0
2.00-1.00	22.4	96.0
1.00-0.00	4.6	100.0

D(AVE) 3.21 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



Administrative Notes

T2 (cont'd.)

HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE C3-P5-T2
SOLVENT

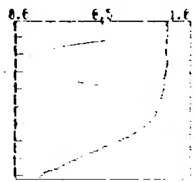
• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.30(G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00(µM)
D(DIV) 1.00(µM)
SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.87

TIME ABSORBANCE



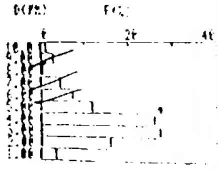
• DISTRIBUTION TABLE (BY VOL.)

D(µM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.2	1.2
9.00-8.00	2.6	3.9
8.00-7.00	0.1	4.0
7.00-6.00	4.4	8.4
6.00-5.00	7.3	15.8
5.00-4.00	11.8	27.5
4.00-3.00	26.1	53.7
3.00-2.00	27.3	81.0
2.00-1.00	15.0	96.0
1.00-0.00	3.4	100.0

D(AVE) 3.14 (µM)

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-P5-T2
SOLVENT 150

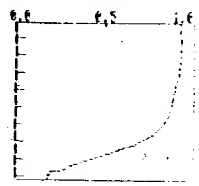
• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.30(G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00(µM)
D(DIV) 1.00(µM)
SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.9

TIME ABSORBANCE



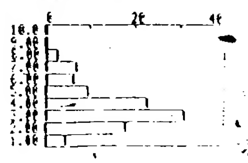
• DISTRIBUTION TABLE (BY VOL.)

D(µM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	2.3	2.3
7.00-6.00	6.8	9.1
6.00-5.00	6.6	15.6
5.00-4.00	9.6	24.6
4.00-3.00	22.5	47.1
3.00-2.00	30.0	77.0
2.00-1.00	18.0	95.7
1.00-0.00	4.3	100.0

D(AVE) 2.91 (µM)

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



Administrative Notes

T1 versus T2 versus T3 for VARIOUS passes

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/23
SAMPLE C3-PI-T3
SOLVENT 150

• CONDITION T3

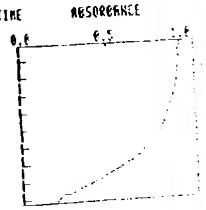
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (μM)
D(MIN) 1.00(μM)
D(DIV) 1.00(μM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~ 0.9

TIME ABSORBANCE



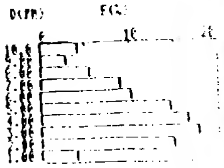
• DISTRIBUTION TABLE (BY VOL.)

D(μM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	4.3	4.3
9.00-8.00	2.9	7.2
8.00-7.00	5.5	12.7
7.00-6.00	9.0	21.7
6.00-5.00	10.3	31.9
5.00-4.00	14.6	46.5
4.00-3.00	16.6	63.1
3.00-2.00	17.9	81.0
2.00-1.00	15.0	96.0
1.00-0.00	4.0	100.0

D(AVE) 3.79 (μM)

• DISTRIBUTION GRAPH (BY VOL.)

D(μM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PIV
SOLVENT

• CONDITIONS 0.75+

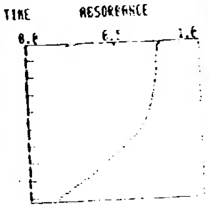
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (μM)
D(MIN) 1.00(μM)
D(DIV) 1.00(μM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA T1

TIME ABSORBANCE



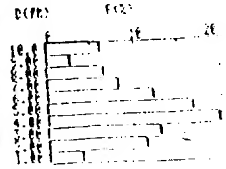
• DISTRIBUTION TABLE (BY VOL.)

D(μM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	6.2	6.2
9.00-8.00	2.7	8.8
8.00-7.00	6.6	15.4
7.00-6.00	8.2	23.6
6.00-5.00	12.0	35.6
5.00-4.00	16.8	52.4
4.00-3.00	19.8	72.2
3.00-2.00	12.8	85.0
2.00-1.00	11.2	96.2
1.00-0.00	3.7	100.0

D(AVE) 4.14 (μM)

• DISTRIBUTION GRAPH (BY VOL.)

D(μM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE C3-PV
SOLVENT

• CONDITIONS NO CHANGE
MILL CONFIG REQUIRED

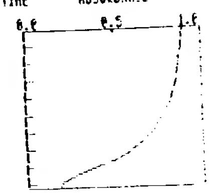
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (μM)
D(MIN) 1.00(μM)
D(DIV) 1.00(μM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA T2

TIME ABSORBANCE



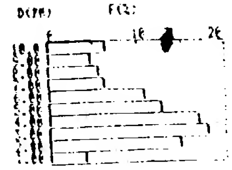
• DISTRIBUTION TABLE (BY VOL.)

D(μM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	6.5	6.5
9.00-8.00	4.7	11.2
8.00-7.00	5.6	16.8
7.00-6.00	6.3	23.1
6.00-5.00	10.7	33.7
5.00-4.00	12.7	46.4
4.00-3.00	16.9	63.4
3.00-2.00	18.0	81.4
2.00-1.00	14.7	96.1
1.00-0.00	3.9	100.0

D(AVE) 3.79 (μM)

• DISTRIBUTION GRAPH (BY VOL.)

D(μM) F(%)



Administrative Notes

FINAL C3-T3- RESULTS : NECK, MIX & premix medium

HOR[illegible]
PARTI[illegible]

SAMPL[illegible]
SOLVENT

• CONDITIONS

SOLV.VISC 2.16(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00(µM)
D(DIV) 1.00(µM)

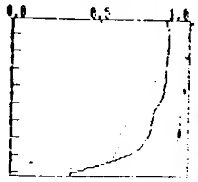
NECK

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE



• DISTRIBUTION TABLE (BY VOL.)

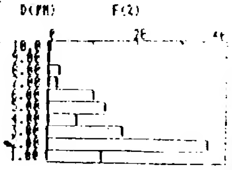
D(µM)	F(%)	R(%)
10.0 <	[illegible]	[illegible]
10.0- 9.[illegible]	[illegible]	0.0
9.00-8.00	0.0	0.0
8.00-7.00	2.4	2.4
7.00-6.00	[illegible]	4.4
6.00-5.00	10.5	14.9
5.00-4.00	13.1	28.0
4.00-3.00	6.5	34.5
3.00-2.00	17.0	51.5
2.00-1.00	[illegible]	87.4
1.00-0.00	[illegible]	100.0

D(AVE) 2.09 (µM)

2

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/23
SAMPLE C3-P3(T3) Mix
SOLVENT T32

• CONDITIONS

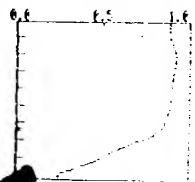
SOLV.VISC 2.16(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00(µM)
D(DIV) 1.00(µM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA D~0.9

TIME ABSORBANCE



• DISTRIBUTION TABLE (BY VOL.)

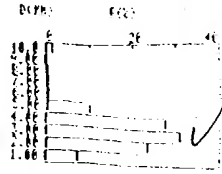
D(µM)	F(%)	R(%)
10.[illegible]	[illegible]	0.0
10.0-[illegible]	[illegible]	1.2
9.00-8.00	[illegible]	1.2
8.00-7.00	0.0	1.2
7.00-6.00	0.0	1.2
6.00-5.00	0.0	1.2
5.00-4.00	5.7	16.9
4.00-3.00	27.7	39.6
3.00-2.00	[illegible]	69.[illegible]
2.00-1.00	[illegible]	92.7
1.00-0.00	[illegible]	100.0

D(AVE) 2.63 (µM)

O.K.

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/25/88
SAMPLE C3-P2-T3
SOLVENT ISO

• CONDITIONS

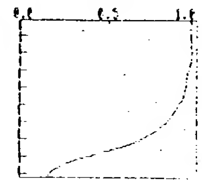
SOLV.VISC 2.16(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (µM)
D(MIN) 1.00(µM)
D(DIV) 1.00(µM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE



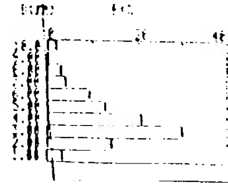
• DISTRIBUTION TABLE (BY VOL.)

D(µM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.8	1.8
9.00-8.00	0.0	1.8
8.00-7.00	3.1	4.9
7.00-6.00	4.3	9.2
6.00-5.00	9.4	18.6
5.00-4.00	12.8	31.5
4.00-3.00	20.7	52.2
3.00-2.00	30.0	82.2
2.00-1.00	14.3	96.5
1.00-0.00	3.5	100.0

D(AVE) 3.11 (µM)

• DISTRIBUTION GRAPH (BY VOL.)

D(µM) F(%)



ATTACHMENT C

YU 10179

IBM

Technical Notebook

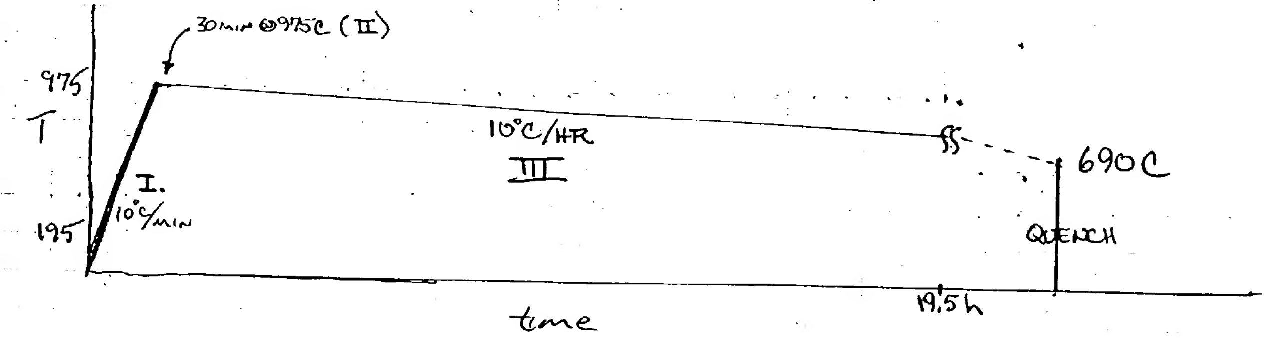
Book V

User's Initials and Last Name:		
DUNCOMBE, P.		
Employee Serial: 155139	Date of First Entry: 6/7/88	Date of Last Entry: 5/89

Security Classification:

6/9/88

Next "transition" Heat Treatment (B)



I. 25C-975 → Δ950C/10C/min = 1.583 h (1h 35min)

II. 975C for 30 min = 0.5h

III. ~~975~~C - 690C = ~~Δ~~285C/10°C/h = 28.5h

total: 28.5 + 0.5 + 1.58 = 30.58 h

Proposed: purge till 10 A.M. Monday
10 - 11:35 AM. → heat up (RAMP I)
11:35 - 12:05 pm → dwell
12:05 pm - 4:35 p.m. Tuesday → RAMP 2 (cool)
4:35 p.m. Tuesday quench

(A) As B above w/ 650C QUENCH w/ I: as above
II: as above
III: 32.5 h
} ~34.5 h total

ON @ 11:40 A.M. 6/13
START RAMP down @ 1:45 p.m. (actually 2:05)
Drop to 946 @ 10C/min then slo ramp to 0.17C/min

Estimated Quench time 25h 24min OR 3:36 p.m. 6/14/88

6/14/88 ⇒ ~~2:45~~ updated QUENCH TIME 3:15

tidbit : (CONVERSION)

$$\text{mm Hg} = 0.001333 \text{ bars} \quad \therefore \quad 0.001333 \text{ bar/torr}$$
$$\downarrow \quad = \quad 1 \text{ torr}$$

$$1.333 \text{ mbar/mtorr} \rightarrow 0.75 \text{ mtorr/mbar}$$

Date and sign every entry. Have every p... y important entry witnessed. Submit an Invent... Dis... re of anything possibly new and invent...

☐ Unclassified
☐ IBM Internal Use Only
☐ IBM Confidential

☐ IBM Confidential-Restricted
☐ Registered IBM Confidential*
*Register with local Recorder

}100, 10°/min, 1550 [redacted] 61 min, 10°/min, 100C } RUN [redacted] SPECS

6/29/88

IBM Technical Notebook

T	PV	PROG 100	MAN%	Amps	Volts	f meter 6.0	ΔP (MIN)	Comments START
12:20	986	990	10.4	490	2.5	6.92	·	4×10^{-5} torr
12:42	1196	1200	14.9	600	3.1	7.11	·	5×10^{-5} torr
12:57	1346	1350	20.4	700	4.0	7.27		4.6x / 12 psi
1:07	1445	1450	25	725	4.25	~~7.37~~		3.5
1:17	1550	1547	30.3	700	5.4	7.48	60.1	↓ 12
1:48	↓	1550	29.7	700 700	5.4	7.47	80	1.6/ 12
2:18	1550	1550	29.2	690+	5.25	7.469	0	1.25/ 3
2:32	1415	1407	20.3	625	4.25	7.34	X	8×10^{-6}/2
3:45	679	678	3.6	300	1.5	6.61	–	2.8E-6/2
4:20	333	325	0	0	0	6.29	X	2.8 / ✓
4:45			0	0	0			

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4/7/5/88 STBX-2 **IBM Technical Notebook**

Time	PV	Prog	Man%	A	V	gauge ?	ΔSP	Comments	Vac
10:40	341	341	3.4	350	1.5	7.76		bias changed @ start	
	640	641	5.2	390	1.6	7.98		20 PSIG applied	6x10^-5
11:42	962	961	86	460	2.4	8.20		40 ↓ ↓	1.8E-5
1:00	1650	1650	35.4	735	6.0	8.88	112.0	✓	1.9E-5
2:30	↓	↓	35.3	725	↓	8.86	22.8	∴ 2:52	2.5E-5
3:00	1576	1565	28.0	700	5.5	8.78	—	→ 2:53	1.2E-5
4:46	532	530	2.5	360	1.75	7.87	— 1.6E-6	load removed (3 PSIG)	4E-6
5:07	~300	~300	0.8	<300	<1	7.70	→	increase flow to 8.5 gpm from 7	
5:10								STOP STP 1.6E-6	
5:15	200	228	off	—	—	7.61		SHUT OFF H2P	
5:20	149	162	—		—	7.52		vacuum constant ~12.5 PV/min; SHUT OFF STP Power,	

started backfill (5:22 STP on mech gears, light out), mech shut down, high vac closed.

5:25 178 134 GAS has RAISED temp, but Ar GPM up to 18.
5:30 190 100 Prog off

Notes: 300 C @ 10:36 ∴ 2h 15m to S.P. ⇒ 12:45 (est) + 2h ⇒ 2:45 Rampdown, ~3.5 hrs for cooling, < 100C opening @ 6:15 approx. (due to thermal mass lag.

Unsuccessful. Same sticking to bottom foil. Cracked with diffusion zone and multiple phase boundary.

The above understood and witnessed by Date and Date

6/28, Bi/Cu

Ar/H_2 Bi 20 { Bi 25 pieces remaining after slicing isostatically pressed to 28,000 PSI OK

Bi 25 → some obvious large void improvement on at least the 1/2

Bi 20 → possible visible evidence of compression, need to section. (especially large 1/2)

Objectives:

1) isopress as above

2) slice

3) anneal remaining sections

4) study wetting/densification vs temperature relationship

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6 7/7 STBX-3

IBM Technical Notebook

Time	PV	Prog	M%	A	V	quadr	ΔSP
~11:00	462	467	35	350	1.5	7.69	--
11:58	1064	1006	9.2	490	2.5	8.23	
1:08	1576	1575	29.5	696	5.5	8.82	22 116.3
1:25	1575	1575	29.3	↓	↓	8.81	91.6
2:45	1575	1575	29.2	675	5.5	8.80	15
4:45	599	599	0.0!				

Comments
est. S.P. start: 1:17, 5E-6
1.8E-5
↓ est. ~3:00 start rampdown
1.0E-5 ⟷ 9.6E-6
7.5E-6

different than last run, even on 7 gpm!

7

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Date

and

Date

7/11 2050 Bake out Pre Al_2O_3 : I88 [PROG 8]

Time	PV	Prog	M%	A	V	Meter	MIN ΔSP	Vac	Comments
12:30	1660	1687	40.1	760	6.4	—	—	5E-5	RAMP 20°/MIN ∴ 1200C/h Some degassing > 1650
12:42	1893	1935	66.2	900	8.4	—	—	4E-4 !	7gpm
12:52	2037	2050	88.7	950	9.5	—	10/5	7E-4	definitely baking out' top was 2040 (back 5 min by)
12:55	2028	2003	84.4	✓	✓				Hold to continue B-out
1:00			92.0						
1:04	2061	H	96.0					4E-4	Raised manually
1:11	2083	H	98.0						PV steady ↓ again
1:17	2094	H	↓	1000	10	—	—	3E-4	Holding
1:34	same							1.9E-4	Vac increasing as hoped
2:05	2092	same						9E-5	
2:30	2093	switched back to Auto						6E-5	Start PV ramp down
	2093	2000							
2:46	1727	1691	38.9	790	7.5	—	—	2E-6	Good VAC for 1800C RUN
3:20	1004	1000	6.7	475	2.5	—	—	9.8E-7	
3:45	572	520	1.0	200	0.0			8E-7	
4:10	255	off	—	—	—	STP300 off		7.5E-7	up to 19gpm from 7
4:30	138								
4:33		Argon flush after ion shut off						3.5E-4	STP shut down to mech bearings
4:38		mechanical off							

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SET-UP FOR I88 Al_2O_3 BXSTAL

SEG	T (°C)	time Duration	Ramp Rate (°C)	SEG time	Psig
01	100°C	0.1		~~1h 60m~~	apply initially: 100
02			+~~600~~/h 566.7	~~2h 50m~~ 3h	
03	1800C	5.1		5h	
04			60 - ~~72~~/h 80	6.7h ~~24h~~ 160	Remove load @ start ramp
05	1400				
~~06~~	~~100C~~	0.1	80 16.25	21.25	
				NET ~~20~~h save 23 total	
07	100C				

Time factors: start @ 10:00 A.M., (after loading & pumping) SEG 04 begins ~5:00 p.m.
SEG 05 ends ~5:30 p.m. next day O.K.

Pressures from 2/13/87 (Book II pg 49 notes)

$$P_s = \frac{Area_{(ref)}\,(P_g)}{A_{spec}}$$: ref - ram xsection (4.9") P_g - gauge spec - specimen

Psig of 180 for Bxstal Run 87III resulted in yield of moly stag and XSTAL indentation. So, in lieu of carbon stage switch not possible at moment, will approx. halve pressure to 100 psig and apply load initially, releasing after ramp down begins. This worked fairly well with Duane's $SrTiO_3$ and similar 87III run yielded only partial cracking of samples.

$$P_{sample} = \frac{(4.9)(\cancel{180}\ 100)}{(0.22)} \cong 2\cancel{440} \sim 2225$$ versus article (ref) max of ~4200 (60%)

10/7/14 I88 Bxstal Run IBM Technical Notebook

Final Prog.

SEG	T	t duration	°C Rate	seg time	Psig
01	100°C	0.1h		6min	110
02			600/h	2.8h	110
03	1800C	4.75h	–	4.75h	110
04			80/h	21.25h	Nom.
05	100C				

1.4-4.0E-5 torr (03)

NOTE: intermediate 60/h ramp skipped also, however temp fell from ~1790 to 1730 in switchover before 80/h ramp kicked in. 4E-5 – 8E-7

Run notes: @ ~7:00 pm 7/13 TC leak noticed during inspection. Repaired as possible. Seems to have taken care. See note above.

Results: Severely cracked xstal. Some sticking, but no rxn. with Mo shim on stage, but carbon/Mo rxn from top ram/shim couple. Pitting/roughing top surface & likely initiating cracks.

Wrap-Up George's Bi/Cu work

Try 10g batch

2.5g Bi 7.5g Cu use

2.48 tare
12.49 after transferal

Also took 1/2 20 { 25 Bi Ar/H2(5) run isostatically pressed forms and will anneal while sintering bar.

To temp @ 3:00 p.m. 7/25/88 18h → 9 A.M. 7/26/88

4:20 p.m. 7/25/88 → new type D {W25Re/W3Re} thermocouple seems stable as well as 818 programmer @ 750C

9.919(8) 4.060

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. ______ DATE 7/26/88
SOURCE 25Bi/Cu Bar OPERATOR PRD
TOTAL WEIGHT 13.981 g. OUTGASSING CONDITIONS ______
TARE WEIGHT 4.060 g.
SAMPLE WEIGHT 9.921 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_c 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\dfrac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

P_2/P_3 - 3.5415 3.5429 3.5419

DATA

	RUN 1	RUN 2	RUN 3
P_2	19.457	19.656	19.746
P_3	5.494	5.548	5.575
V_p	1.2006 cc	1.219 cc	1.206 cc
DENSITY	8.2634 g/cc	8.1316 g/cc	g/cc
	8.26/9.17 = 90%	8.14/9.17 = 89%	8.23/9.17 = 89.75

10X

16

Average: 89 + 89.75 ≈ 89.5   Between 89-90%

ENTERED 8/1/88

A. Liq. ϕ Bi/Cu System Development Summary

initial compositions: Conditions: 400C in N_2 overnite

% Bi (liq)	% Cu (solid)
1	balance
3	
10	

NOTES: CONDITIONS AND % comp. Bi did not lead to densification (sintering) of plugs. No weight loss. 5g samples in vitreous carbon crucibles

B. Conditions: 750C in N_2 overnite

% Bi	% Cu
50	balance
10	
(3)?	

NOTES: wetting in N_2 does not seem to be total, possibly oxide films interfering behavior. 50/50 mix too much liq. Bi pockets apparent. 10% too little liq. No appreciable wght loss.

C. Conditions: 750C in N_2 overnite

% Bi	% Cu
25	bal
35	

NOTES: ~ density 25 Bi (90) by 61. fr calc. (assume 9.1g/cc for 25% fully dense body). Definitely beaddating desired range or close. 35% Bi density not calculated but volume obviously smaller ∴ sample density is at least same or higher. Still evidence of incomplete wetting. Mean third β. evident of possible oxide)?

D. Compressed pellet RUN Conditions: 750C in Ar/5H2 overnite

% Bi	% Cu
25	bal

NOTES: ~ calc density 25 Bi pressed (79%) above basis. Evidence of better wetting, supporting oxide assumption stated previously. Low density due to pellet bloating, probably trapped air in pellet. No significant Bi vaporization evident by wght loss.

E. Conditions: 'packed' powder crucible "free" sinter @ 750C overnite in Ar/H_2. No compaction.

% Bi	% Cu
25	balance

NOTES: Porosity is reduced, 3rd (grey) ϕ "oxide" has been eliminated in forming gas, wetting seems approx complete. Little difference between ① <100 mesh spherical Cu and ② 10 μm Cu, though 10 μm seems to give better overall results. ③ Penetration RUN seems to need more time, but wetting characteristics seem better. Will rerun sample in next run (i. 2x overnite)

F. Vacuum 25 Bi Run

CONCLUSIONS: highly porous. Vacuum doesn't appear to work well ~~in Ar/H2~~. No evidence of oxidation, however, indicating vacuum was O.K. No evidence of vapor phase deposition in glass tube.

G. Conditions: 'packed' powder crucible sintered @ 750C overnite in Ar/H_2. No compaction.

% Bi	% Cu
10	balance
20	
25	

Results: Good areas and wetting in both 20 & 25% samples, however 20% seems better overall with small 'can form' pores whereas 25% has some voids as well as pores.

20 & 25% Bi samples pressed to 27,500 psi isostatically. Some collapse of large porosity/voids. Little effect on small porosity.

Annealing effect: 0.02 & 0.04 g of Bi-rich liq. ran out of microstructure upon annealing. Porosity diminished. Very good looking virtually dense 20% sample.

'Bar': 25% Bi (10g batch) in vitreous boat first placed in house vacuum (dessicator) then sintered overnite per std. treatment producing very good looking micro-structure with little porosity.

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8-11-88

Green phase - substrate work

Seet not found from memory

have one remaining substrate, ~80-90% dense, single phase, sinter'T' 1350C

(1) pressed 0.2", 0.2g pellet of eutectic

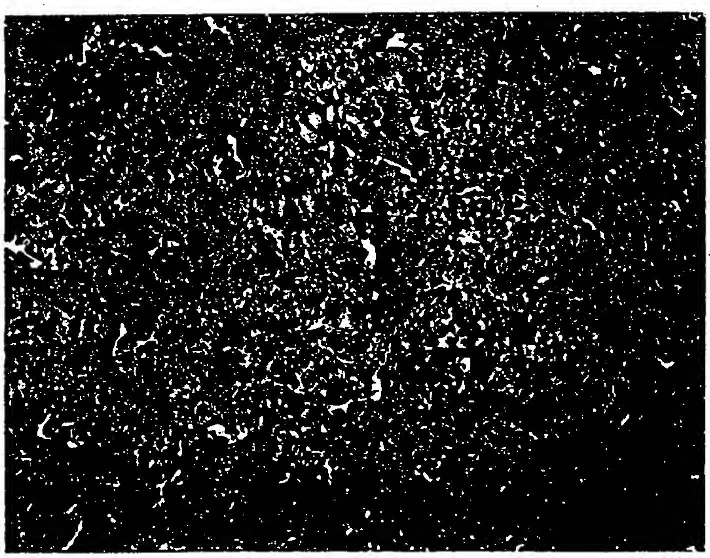
8-18-88 week summary

1500C pellet almost totally melts (2 ϕ) with interaction between Al_2O_3 and liq. ϕ. (211 coarse "off comp")

1400C pellet retains its integrity, but large amount of liq forms 2ϕ, interaction w/ liq ϕ and support

1350C liq ϕ still present, though diminished. less interaction. for short sinter time (211 milled "on")

1300C 211 1 H 100X milled powder.



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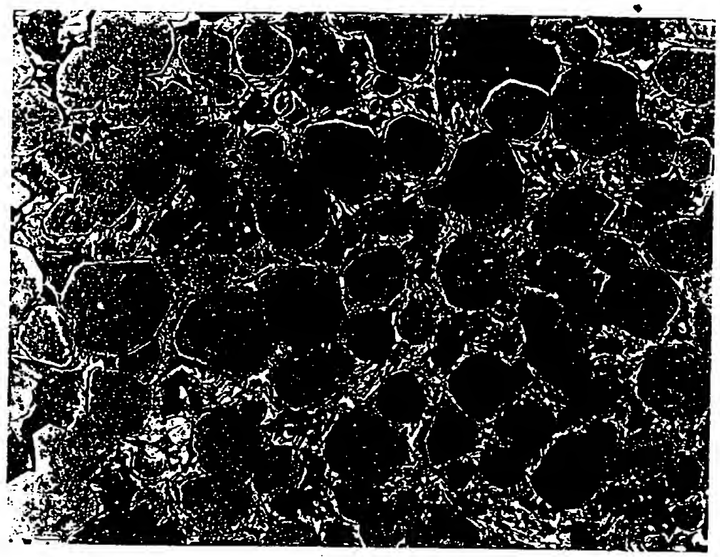
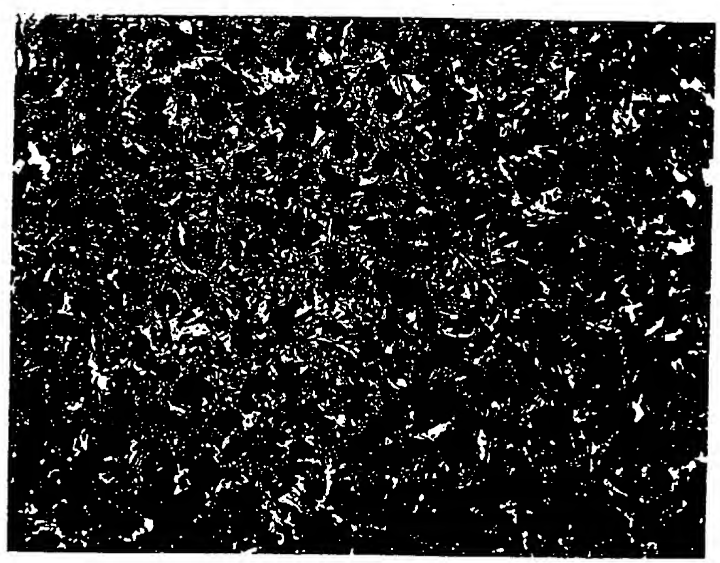
1292C 18HRS 211 milled 100X



1265C 'coarse' 'off comp' OVERNITE 100X



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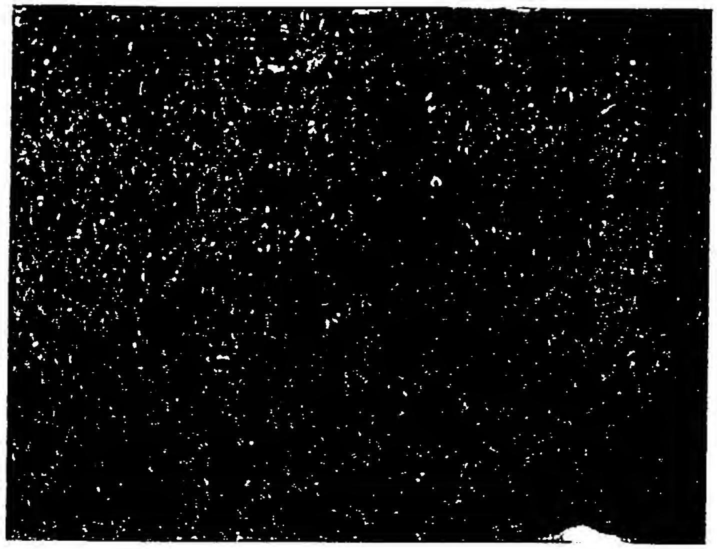
211 milled 1235C 2HRS <u>1000X</u>



Conclusion: sintering @ 1292C or higher creates 2 ø material
exaggerated liq ø grain growth after prolonged period

sintering @ 1235C does not induce adequate sintering.
<u>pellet remains green</u> as opposed to higher temp, where
pellet turns black (presumably this is <u>not</u> simply
surface effect, but has chemical basis)

sintering @ 1265C may be optimal.
Powder definitely ooo?

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Green density - 211M 0.6 pellet
5500

2.56 1.548 0.399 0.7509 3.41 / (~~6.36~~) = ~50%

150
✓ 1.472 0.389 0.662 3.87 / (~~6.36~~) ~ 60.9
(6.00) ~64.5

Post → pellet not good enuf to bother. 2φ, stuck, etc. (1292C) 18H
1265C pellet II (150 29)

2.56 1.457 0.398 0.66 3.88 / (6.00) ~64.5

pellet cracked on checking must REDO, TEMP O.K. though (Gain 5C)
pellet III in furnace @ 4:10 to temp @ 4:30
set 1255, Tsample ~ 1270

2.53 1.286 0.360 0.47 5.38 / (6.00) ~ 89.7%

8/23/88 15029, pellet IV (second 'good')

2.81 1.455 0.438 0.7283 3.86 ~64.3%
8/26
2.8 1.283 0.384 0.4964 5.64 ~94

15030, pellet V (edge chipping during wopressing) O.K.

2.94 1.456 0.457 0.761 3.86 ~64.4% consistent

4:20 to temp @ 1267C

2.93(5) 1.283 0.4– 0.517 5.67 ~ 94.5 ~65h
~~2.9~~
3 good slices

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8-31

0.04g 0.33mm dia pellet set on edge of polished 211 substrate which itself rests on a piece of 211 resting in a Al_2O_3 boat on a bed of 123. Adjacent to substrate is small pellet of 211 to allow eutectic pellet to straddle edge of substrate to minimize contact.

Heat treatment: 10°C/min to 1000C in flowing O_2

previous exps. in air/O_2 showed incongruent melting of eutectic @ ~1000C.

10:45 A.M. T @ 500C ∴ 1000C plateau should be reached @ 11:35

Will allow to melt for 1h → 12:35

10C/min → ~~785~~ 1:00

5C/min → 600 1:30 hold

~~10C~~h → ~~quench 2:30~~

→ 10C/min → 300C quench

Flow not pronounced. Not alot of liq. formation. Pix taken.

Redo in Air/O_2 where prev. exp. showed alot of liq. formation.

2.[illegible]

9/6
$Bi_{2.15}\ Sr_{1.68}\ Ca_{1.17}\ Cu_2\ O_{8+\delta}$ (X-RAY single φ)
2 2 1 2

$Ca_{(2-x)} Sr_x Cu O_x$ ← Nature letters $(1-x)$ $Ca_{0.86}\ Sr_{0.14}\ Cu_1\ O_2$
$Bi_2\ Sr_2\ Cu\ O_x$ 2.1 1.6 & 2.2 1.6 so use 2.15 1.6 1.0

Bi	208.980	Bi_2O_3	465.9582 g/M	2 moles!
Sr	87.62	SrO	103.6194	
Ca	40.08	CaO	56.0794	
Cu	63.54	CuO	79.5394	

2.15 moles	Bi_2O_3	1.00181013 g X 150	⇒	150.2715195 "2 moles"
1.68 ↓	SrO	0.17408059 mg		26.1120885
1.17	CaO	0.06561290 mg		9.841935
2.0	CuO	0.15907880 mg		23.86182
				210.0871363 g

CO_3 - 60.00935
$SrCO_3$ - 147.62935 $CaCO_3$ - 100.08935

$\frac{SrCO_3}{SrO} : \frac{147.62935}{103.6194} = 1.42472693\ (26.1120885) = 37.20259576$

$\frac{CaCO_3}{CaO} : \frac{100.08935}{56.0794} = 1.78477926\ (9.841935) = 17.56568146$

(1) Mixed, ground and calcined @ 775C 21 h (PE)

(2) Grd and kept in Al_2O_3 boat covered w/ Au foil @ 800C for 6 h

(3) Grd and recalcined @ 850C for 16h

(4) Grd & mill for pelletization

Factor of 2 molar correction

150.2715195 /4 = ~~37.56787988~~ 18.78393994 g

37.20259576 9.30064894 (0.7018889) = 6.528

17.5656846 4.39142037 (0.56029338) = 2.4605

23.86182 5.965455

57.22540 ~~(0.7018889)~~
− 1.931
− 2.7726
52.522 g batch

scale up everything by 1.5

28.176
13.950 ⊗
6.587 ⊗
8.948
57.661 (less CO_2 loss*)

13.95 (.7075) = 9.7895 4.16 g loss
6.59 (0.560) = 3.69 2.9 g loss
7.06 total expected

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9-7	calc'd	wgh'd	GRAVREC	
B_2O_3	28.176	28.18	207.28 ← bottle tare; 235.45; 28.17	post 207.30(1); Δ + 0.03; .03/57.7 < 0.05% loss
SrO	13.950	13.96	235.45; 249.41; 13.96	
CaO	6.587	6.59	249.41 (2); 256.02; 6.61 + 0.01(2)!; − 0.01(2); 6.60	recovery better
CuO	8.948	8.96	256.00 (0.02); 264.96; 8.96	

(1) Powdr. transferred to O.D. tall bottle, shaken for > 15 mins.
(2) to 400ml beaker w/ ~150ml (made up to) ~~46~~ 150
(3) Continuously stirred w/ mag. stirrer while removing solvent. 11:30 → 1:20
(4) Stirrer removed lowered to 'low', dry for oven @ 2:00
In oven under vac @ "3" 2:05 → 3:45

146.59(8)
89.12
57.47 vs 57.69 (0.4% loss) ~~may be some~~

beaker 159.46

152.23(2) w/ top

146.59
142.15 post 20 NO EVIDENCE OF VAPOR
4.44 g loss ⇒ 7.06 g expected ∴ 63% conversion if no Ba loss
89.12
53.03 ~51.26 52.86 ... 99.7 % > Post grd 51.43 97%

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24 9/9

IBM Technical Notebook

BiSrCaCu Calcination II : gold lined Al_2O_3 boat

86.51 barely fits in larger boat
35.13

51.38 vs 51.43 0.1 % transfer loss

85.10
35.13
49.97

49.34 IN furnace (tube) for 850C, 16h calcination

84.50(48)
.21 stuck in screen
84.71

83.58 post 850C 16h calcination
35.13
48.45 need on X-RAY

9-7 $Bi_{2.15}\ Sr_{1.6}\ Cu_1\ O_x$ (ref. data pg 21)

2.15 mmoles Bi_2O_3 1.00181013 but $2Bi = 1BiO_2$ ∴

Bi ∴ 0.50090507 mg/mM

1.6 mmoles $SrO \to (1.6)(103.6194) = 0.16579104$ mg/mM

1.0 mM $CuO \to$ = 0.0795394

$SrO \to SrCO_3 \to 1.42472693(0.16579104) = 0.2362(0696)$

scale factor for 50g lot ~60

(60) (0.50090507) = 30.0543 Bi ox

(0.23620696) = 14.1724 Sr as $SrCO_3$

(0.0795394) = 4.7724 Cu

48.9991 ~ 49g ox close enuf.

10-17-88 MISTAKE ← NOT Applied

$Ca_{0.86}\ Sr_{0.14}\ Cu_1\ O_2$

0.86 (56.0794) = 0.048228284 (1.785) = 0.0861

0.14 (103.6194) = 0.014506716 (1.42472693) = 0.02066811

1.0 (79.5394) = 0.0795394

scale factor for 50g batch (340)

340 (0.048228284) = 16.398 — 29.27

(0.02066811) = ~~4.932~~ 7.027 (4.949) — 7.028

(0.0795394) = 27.043 — 27.04

50.468 g (less CO_2) — 63.34

2.078

48.39

$SrCO_3$ decomp. @ 1340°C
Bi_2O_3 melt @ 880°C
$CaCO_3$ decomp 825°C

CuO - 1026C decomp,
all below initial calcination T!
775 C

9/13 from Chandra:

$Bi_{2.15}Sr_{1.6}Cu_1O_x$ procedure for calcination - all Pt.

752C for 6h ← GRD. ✓
790 C overnite (16h)
~~(800C)~~
825C 16h NOT CONVERTED
890C < 855 20h

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1423 - 273 = 1150 | 800C - 825C

27

9/8 $Bi_{2.15}Sr_{1.6}Cu_1O_x$

①	calc'd	wgh'd		
Bi_2O_3	30.0543	30.06	201.50 tare / 231.55 / 30.05 ✓	
$SrCO_3$	14.1724	14.18	245.72 / 14.17 ✓	(9.98 oxide)
CuO	4.7724 / .999 = 4.777	4.78	250.50 (49) / 4.78 ✓	OK.

$Ca_{0.86}Sr_{0.14}Cu_1O_x$ ←

$CaCO_3$	16.398	16.40	211.32 (3) tare / 227.74 (2-6) ③ / 16.42 recovery	slightly unstable
$SrCO_3$	2.027	2.03(4)	229.79 / 2.03	
CuO	27.043	27.04	261.81 / 27.04 ✓	OK.
	27.043/.999 = 27.07		.03 added / 27.07	

Shaken, suspended in iso, mixed & dried as per 2212 previously.
Will start both tomorrow after consultation w/ R. Fagat.
Merrill says try 800C to start in Pt.
No - transferred to HCl cleaned BiSrCaCu crucible & fired for 6 h overnite.

137.87
89.15 tare (89.12) 0.03 exp
48.72 / 49. expected 0.28/49 =
135.86 Post 6 → Greenish yellow (46.71)
−Δ 2.01 (/4.19 expected) 48% reacted

135.51 (46.34)

The above understood and witnessed by ______ Date ______ and by ______ Date

133.28
133.66

Calcination II (post grind #1)

BiSrCu

135.51
89.15
46.34 -Δ 0.37/46.71 (0.8% loss)

133.66 post 790C 16h brownish-black hue, little sintering
89.15
44.51

44.10 let stand overnite, will run x-ray in morning.
132.66 post 825C 16h rich black texture, little sintering
exterior of sinter mass (tan?)

CONT pg 30 →

Calcination II SrCaCu ~~from pg 23~~

140.92
90.48
50.43 /50.52 0.03% loss

16.42 (0.56) = 9.1952 (-Δ 7.225)
7.03 (0.702) = 4.935 (-Δ 2.095)
−9.32

50.52
−9.32
41.20 ∴ 41.2/42.8 96%

133.28
90.49
42.79

42.43 post grind 0.8% loss. {Product looked good, little sintering, black.}

132.90
90.49
42.41 In furnace for 16h @ 1000C. Partial melting.
9/13 Run aborted. Restart. Cal I @ 800C then x-ray.

9/13 $Ca_{0.88}Sr_{0.14}Cu_1O_x$ RESTART // melting @ 1,130 want 1,000 IN experimental synthesis

XSTALS excited Chandra. Same formula over.

9/14

	calc'd	weighed	
$CaCO_3$	16.398	16.41	227.76
			211.35 (4) tare
			16.41
$SrCO_3$	7.027	7.04	234.80
			7.04
CuO	27.07	27.07	261.84 (5)
			27.04 (5) ✓

9/15 Run shot due to bumping when someone turned up heat

#3

$CaCO_3$	16.41	2
		211.45
		227.85 (6)
		16.40 (1)
$SrCO_3$	7.04	227.86
		234.90
		7.04 ✓
CuO	27.07	261.96 (7)
		27.06 ✓✓

#4 - 9/21/88

$CaCO_3$	16.41	211.49 (±1)
		227.89
		16.40
$SrCO_3$	7.04	234.92 (±1)
		227.89
		7.03 ✓
CuO		261.98 (9)
		27.06 ✓

One last time 9/21

IBM Technical Notebook

Gold for Dick
22.87 g rec'd
8.92 owed
13.95 remaining

8.92
5.88 not used
3.04 returned unused

$CaCO_3$ 16.41 — 227.88 (9) / 211.48 ~~211~~ tare (+1) / 16.40

SrCO 7.04 — ~~234.93~~ (13) / 227.89 / 7.04

CuO 27.07 — 262.— (61.99) / 234.93 / 27.06

VACATION ⇒ 9/26, 25, 24, 23, 22/88

After drying (~days) weekend

138.94
89.15 (14)
49.79 g Recovered / 50.5 0.71/50.5 1.4% (bumping, etc.)

VACATION 9/28, 29, 30/88

POST I
138.94
131.15
7.79 loss

131.15
89.15
42.00 g net

II finish
40.3 g collected after grinding for possible cal III

TO PAGE 32

PRE II
131.07
89.17

POST II
129.66
089.16
40.00

40.52

9.79 g loss

Date and sign every entry. Have entry witnessed. Submit an [redacted]r, possibly important anything possibly new and [redacted]ive. Disclosure of

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$Bi_{2,0}$ $Bi_{2,1}$ $Bi_{2,2}$

9/14

43 gr $Bi_{2.15}Sr_{1.6}CuO_x$ collected after 16h 825C cal. {<100 mesh gr
will await X-Ray tomorrow morning
Set-up jet mill. Dick still busy on X-Ray
9/19

10/3

Definitely NOT CONVERTED! X-RAY RUN.

10/4

Calcination II - 850C 20h

Pre 132.19 Post 132.03 36h total: significant sintering has occurred
89.12 89.12 probably "went". MINOR sticking
43.01 to crucible (Pt).

38.93 recovery

10/6

Cal III 875C 16h Sintered. Not very hard but has "metallic" luster. No melting.

(10/7) FINAL cal. X-RAY shows some small additional peaks, but predominantly "2201"

The above understood and witnessed by ______ Date and by Date

NOMINALLY

10/4 900C melt test on partially reacted CaSrCu : "0011"

NOTE: after 875 16h pattern discernable in x-ray but multicomponent MIXTURE. ABOVE TEST TO SEE IF 900C cal safe

g
126.92
86.66 tare
40.26

890C seems to be temp (+5C)

10/7 Chandra says 880C may be onset. Final cal. temp suggested @ 865C

10/6

925C 16h : X-Ray shows

10/7 965C ~~16~~ h

126.37
86.66
39.71

appears as usual, some minor sintering.
Still reacting, pattern loosing other Ø peaks and increasing intensities of 0011.

966C ~60h

Swith to 810 when 812 went wild.
One overshoot to 1006C for < 1 minute.
> Doubt if sample saw it.
Removes, some Pt exp on 1 side, harder than usual, reground & replaced for weekend ster. Hope for the best.

$P_b - 6$ $t_i = 30$ $t_d = 10$ } optimization parameters (temp.)

9/15

14.04 / 4.06

Stereopycnometer density 2212: 6.45 g/cc

	g	t mm	diam mm				
I	0.21(5)	.211	.538	0.0462	4.65	72%	too high if correct
PRE II	0.27	0.283	0.518	0.0599	4.51	69.9%	lower
POST	0.265	0.293	0.574	0.076	3.49!	54	No way. 70h 852-5

875C overnite melted/VAPORIZED

yes! (11-3-88)

Sintering Temps - rapid temp

Tset	Tcont	Tsample		
830	836	858		
830	835	850	equil.	no sintering appreciable
855	863	875	equil.	incongruent melting/VAPORIZATION
835	–	855		
		851	equil.	no melting, looks pretty good

10/3 Analytical Results (ICP)

82.45 total

	wt %	mol %	(theor) MIX mol%	+Δ mol%
Bi	49.0	2.34	2.15	6
Sr	15.8	1.78	1.68	3
Ca	4.85	1.21	1.17	✓
Cu	12.8	2.0	2	–

	m.w.	wt%-mol% conv.
Bi	208.98	~~.234~~
Sr	87.62	~~1.38~~
Ca	40.08	~~0.631~~
Cu	63.54	~~1~~

UNNECESSARY

13.55 theoretical wt% OX @ 2212
82.45
~96.00

 P2 - ~4500 150-28,500 **IBM Technical Notebook**

10/4

1.17 0.095 0.278 0.262 4.465 69%

START SINTERING @ 4:00 PM Rapid temp setting 835. Should give pellet sintering temp of 855.

4:20 840 Tc prompt ⇒ Ts 854 } slightly lower overshoot
846 Tc Read

4:30 839 was Ts 859 Reduced
836

4:50 ↓ Stable @ 856

10/5 8:30 AM 16h sinter check

1.14 1.15⊛ 0.287 0.291 (3.92)

Pellet warped, flowed (⊛approx. dia. due to non-vertical sides) and possibly has second ϕ RXN on part of top surface. Peak temp as far as I saw was 859. MUST keep below 850 (or at). C.

PRE

0.94 1.12 ~0.21 0.21 4.48 69.5 ~OK

pellet looks good @ 852 after 20 h (overnite) Keep sintering for grain growth

10/10

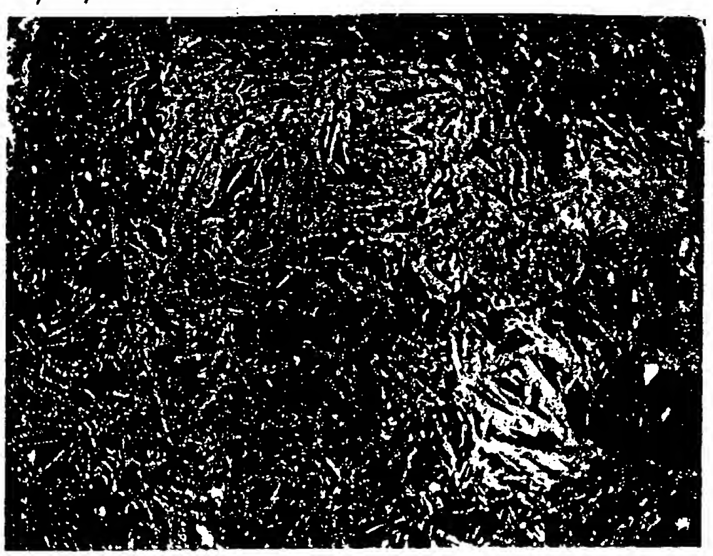
0.90 1.23 ~0.23 0.27 3.3 51% does not make sense

NOTE: T. SHAW says people have seen such effects → same as previous results though

$Bi_{2.15}Sr_{1.68}Ca_{1.17}Cu_2O_{8+\delta}$

"22.12"

Analysis Results Pg. 33



1000X
120h
850C

The above understood and witnessed by:

Date

~~10/6~~ 2212 DYNAMIC SINTERING EXPERIMENTS

Sintering conditions: 850-854°C in Air/O_2: will need to preheat furnace to achieve short duration sintering times.

5 (15) (30) ~~45~~, ~~60~~ minutes @ first

10/13 Pre
1.29 1.093 0.311 0.292 4.42 68.5% UNL 3,750 ISO 27,500

In furnace 10:26 ~750 Tstage 11:04 out (38 overall)
10:30 840
10:33 851 Start TΔ sinter count (855-852)

Post
½ h 1.27 1.142 0.326 0.33 3.85 ~~60%~~ → 59.7

15 MIN Pre
1.19 1.095) ~0.288 0.27 4.41 68.4

In furnace 12:52 ~800 Tstage (plate cool)
(20 overall) 12:56 850+ (pumping by upto Tset 865)
12:56 850 ~852/3 Δ15 ⇒ ~ 1:12
1:01 854

Post
1.17 1.131 ~0.296 0.297 3.74 ~~61%~~ → 61.1

Pre ~(1.1)
(5) MIN 1.13 1.098 0.27 0.257 4.4 68.2
Post
1.11 ~1.108 0.271 0.261 4.25 65.9

2:32 → IN
2:34 → START } 853
2:39 → OUT } ±2

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10-13

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1.2 g irregular pellet of precip "0011" isopressed to 27,500 psi and sintered for microstructural investigation @ ~972C.
> Powder does not pelletize well @ all.

~2.h @ 972C 2ϕ

Post "2201"

1.21g 1.045 0.233 ~0.2 6.05 2hs 875 (peak 880C)

6.05/7.20 = 84%

NO PRE DATA

#2201-1 11-14-88

Post polishing data recheck

1.04 ~1.046 0.200 0.172 6.05 2hs ✓

The above understood Date and Date

10-17

0011 Synthesis

	theor.	weigh'd	
$SrCO_3$	7.027	7.04	if 0.88 / 6.03
$CaCO_3$	29.27	29.28	29.95 (Δ = 0.68)
CuO	27.04	27.05(4)	

$CaCO_3$ 240.77
tare 211.48
29.29 ✓

CuO 267.80
240.77
27.03 ✓

$SrCO_3$ 274.85
267.80
7.05 ✓

after mixing & drying: 63.15 / 63.37 = ~0.3% loss

Pre CAL I
149.62(1)
tare 86.46(7)
63.15 ✓

Post 16h 875C
134.56(7)
86.46(7)
48.10

29.29 (0.5603)[x] = 16.41
7.05 (0.7019)[x] = 4.95
27.03 (1) = 27.03
48.39 total

[x] pg 21

w/ top 188.03
→ 38.42 (Add to audit)

CO_2 conv.: 48.1/48.39 looks complete

Pre CAL II (47.84)/48.1 = 0.5% grinding loss (to temp (966 @ 4:00 p.m. 10-19-88))
134.47
86.63
47.84

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10-19-88
~~38~~

IBM Technical Notebook

Note: 810 optimized process parameters

$P_b = 4$ $t_i = 15$ $t_d = 5$ $\Delta P = 2.0$

Bi Synthesis Series #1

Variances

	2201	0011	2212
Bi	+3%	—	+8.84%
Sr	−6.25%	−7.1%	+5.95%
Ca	—	−4.5%⊛	+2.6%
Cu	✓	✓	✓ †

⊛ estimated: 0.821/0.86 0.46 (1.215)   †: normalized to Cu

	Anal / Theor		
Bi	2.22/2.15	—/—	2.34/2.15
Sc	1.5/1.6	0.13/0.14	1.78/1.68
Ca	—/—	0.82⊛/0.86	1.2/1.17
Cu	1	1	2

ANALYTICAL Results

	2201 act.	theoretical	0011 act.	theoret.
Bi	2.21(5)	2.15	—	—
Sr	1.5	1.6	0.13	0.14
Ca	—	—	0.46⊛	0.86
Cu	1	1 ✓	1	

⊛ Note: Ca concentration off due to omission of oxide to carbonate conversion factor in initial calculations. This is consistent with large amount of CuO second ϕ.

2212		
Bi	2.34	2.15
Sr	1.78	1.68
Ca	1.2	1.17
Cu	2.0	2 ✓

The above understood Date and Date

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10-26 0011 milled (10/25), X-Ray indicates ~ single ø

die body : 0.483" / 1.228 mm I.D.

@ 8,500 psi pwde press too fragile to go in iso. left after afew attempts @ pressing w/ resultant crumbling.

Next time : ~ 3,600 psi ⟹ 16,000 may need to remill, PSD not available presently.

Pre data not taken;

POST : 875C for 3h (peak-5min-@886C) Rapid temp find at 948

1.36$^+$ (pellet damage ⟹ ~1.4) 3.68/~~4.86~~ = 75.6 ⟹ 76. (damage)

1.174 0.352 0.381 cc 3.675 NEED pycnometer density.

Sintered microstructure reveals ~ 80-85% dense pellet w/ minor 1-2% probably CuO phase in some triple points. ~~Grains ... 2 color mottling (light & dark yellow).~~ ⟹ Exp film

1000X, 3h sinter, 0011, POLARIZED

The above understood and witnessed by ____________ Date ____________ and by ____________ Date

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY 4.95 theoretical

DENSITY - 4.87

SAMPLE I.D. Comp. "0011" DATE 10-27-88
SOURCE SYN II OPERATOR PRD
TOTAL WEIGHT 19.02 g. OUTGASSING CONDITIONS N_2
TARE WEIGHT 4.06 g.
SAMPLE WEIGHT 14.96 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_C 34.85 cc

OPERATIONAL EQUATION $V_P = V_C + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_P = Volume of Powder (cc)
V_C = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

3.685 3.69(15) 3.691 3.692

DATA

	RUN 1	RUN 2	RUN 3	Run 4
P_2	19.646	19.831	19.683	19.718
P_3	5.331	5.372	5.333	5.341
V_P	2.999 cc	3.076 cc	3.07 cc	
DENSITY	~~4.99~~ g/cc	4.86 g/cc	4.87 g/cc	

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 2201 DATE 10-27-88
SOURCE OPERATOR PRD
TOTAL WEIGHT 15.04 g. OUTGASSING CONDITIONS N_2
TARE WEIGHT 4.06 g.
SAMPLE WEIGHT ~~3.70~~ 10.98 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_C 34.85 cc

OPERATIONAL EQUATION $V_P = V_C + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_P = Volume of Powder (cc)
V_C = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

3.568 3.566 3.566 3.566

DATA

	RUN 1	RUN 2	RUN 3	
P_2	19.865	19.720	19.807	19.661
P_3	5.568	5.530	5.555	5.514
V_P	~~1.548~~ cc	1.522 cc	1.522 cc	1.522
DENSITY	g/cc	7.21 g/cc	7.21 g/cc	

0011 Rapid Temp Sintering Process parameters

10-31-88

T_{set}	T_{sample} (equil. values)
962	986
950	978
948	977

note: preheated chamber and overshoot T_{set} needed w/ back-offs on approach to get quick sintering temperature attainment

Diffusion Pellet Calculations: DOPSD!

123 std. pellet volume: 3.25g/6.36g/cc = 0.51 cc

∴ 2201 = 0.51 cc × 7.2 g/cc = 3.67 g ~ 3.75 g

0011 = 0.51 cc × 4.86 g/cc = 2.48 ~ 2.50 g

In preheated RT @ 3:31

	Tset	Ts	Tsample		
0011 ~~0011~~	948	-9??	933	3:31	
		956	965	3:33	
	958	964	974	3:35	backing off now to maintain temp. till EQ
	950	956	974	4:20	
	951			4:21	for nite

 11-7-88

Survey 2212 sintering time versus rel density

h	Rel D(%)	wt
0	68.4	
0.08	65.9	0.97
0.25	61.1	
0.5	59.7	
(16) 70	54	
120	51	

small pellet, density determination probably not as accurate

16 h (not listed in book)

POST 0.88 1.15 0.216 0.22 4.0 / 6.45 62

REDO

Sample normalization

~~10~~-2-88 0011 Analytical

El.	Wt %	theo. M%	Anal M%
Ca	22.4	0.86	0.875
Sr	8.24	0.14	0.147
Cu	40.6	1	1

Example calc.

$\frac{Cu\ wt\%}{Cu\ MW} = 0.639/.639 = 1$

$\frac{Sr\ wt\%}{Sr\ MW} = 0.094/.639 = 0.147$

$\frac{Ca\ wt\%}{Ca\ MW} = 0.559 = 0.825$

11/3

0011 pellet 2 for 16h diffusion sinter

PRE 4,000/30,000 slightly irregular

2.85 1.531 0.496 0.913 ~3.12 /4.95 = 63 — perfect

2.81 ~1.36 0.44 0.639 0.64 4.4 = 89

0201 4,000/30,000 PRE

3.78 1.365 0.494 0.723 5.23 = 72.4 % (too high?)

11/4

0011-2 cut into 2 slices. Didn't add blade thickness so unequal.
1 ~.230 cm thick 1 ~ 0.179 cm
will use for first press

Post

875C for 30hrs ⇒ pellet has warped, grown large nodule like xstals and sagged. Obviously metastable.

previous 3h sinter showed no evidence of instability.

~~11-28-88~~

11-9 2201 pellets

201-3	2.99	3200/28 1.351	0.415	0.595	5.02	69.7	open porosity
	2.96	~1.306	~~0.372~~ 0.37	0.498	~5.98	~ 83.3+ defects	PYCNOMETER 86

2-3 MIN 872C SINTER (SLO ATTAINMENT 2 MIN VERSUS 1 ∴ $T_{SINT} = T_{AT} + 3$ MIN)   air = 85

201-4	3300/30 1.16	1.082	0.245	0.225	5.16	71.7	5 MIN.
	~~1.15~~	1.050*	.210+	0.182	6.3(2)	87.5	double
201-5	1.17	1.086	0.248	0.220	5.09	70.7	broken before sintering

Reground Repressed } pellet 201-11

201-6	0.99	1.094	0.205	0.193	5.13	71.25	to temp 1845
*+	0.98	1.06	0.185 lip	0.163	6.01	83.5	out ~~2:15~~ 30 MIN
*			~(0.178)	0.158	6.2	86	

*(+): 201-4,6 some evidence of drooping  edge droop in pellet. Reduce temp 5C

201-7	2700/29 1.2	1.086	0.258	0.238(5)	5.03	69.9	to temp 2:27-8
1.01 1.06	1.19	~~1.06~~ 1.05	~~0.185~~ ~0.225	~~0.163~~ 0.195	6.1	84.7	~~2:45~~ 15

11-10 Sintering Summary <2201 data>

201-2

	temp	sinter t	green	post	
201-8	872C	2 min	71.25	85.6	← 011-2201 P3 pressed pellet (large die)
201-3	872	2-3	69.7+	86	
201-4	875C+	5	71.7	87.5	86.1 ave
201-9	872C	5	71—	84.7	
201-7	872C	15	69.9	84.7	
201-6	875C+	30	71.25	83.5	
201-10	872C	1h	70.4	~~86?~~ → 85	
201-1	875+	P.D.S. 2h		84—	
201-2	875	~~10~~ 30h		~~(76.1)~~ → 79	

Record keeping: 201-2 30h 875C ~75% (pyc): irregular pellet growth resulting in varying local densities
201-3 0.608 dia. pellet for pressure diffusion sinter
201-5 regrd → 201-11

11-11 GAS pycnometry gives an averaged rel density for pellets 1,7,8,9 (wght 4.6g) of 86.75g vs 84.75 (reasonable agreement), mostly closed porosity.

~~41~~ 46-14 SINTER 870-875C

NOTE: [ALL SINTER TIMES ARE 1 MIN attainment ? / 1 MIN: EQ SOAK + Δ SINTER time]

201-8 perfect pellet ~3000/29000

	1.19	1.081	0.253	0.232	5.13	71.25	green
POST 2 MIN	1.17	1.038	0.225	0.190	6.16	85.6	
201-9	1.2	1.075	0.259	0.235	5.11	71.00	green
POST 5 MIN	1.19	1.036	0.231	0.195	6.10	84.7	
201-10	1.10	1.083	0.236	0.217	5.07	70.4	see below
POST 1h	1.09	(1.043-1.07) ~1.057*	~0.2†	0.175(5)	6.21	~~86.25~~	(accurate?)

201-11 Pellet was reground & repressed from broken pellet. Also, die RAM froze resulting in much high UNIAXIAL pressure (of true edge) 12,000

1.169 0.252 0.27 5.04 70.08

* linear average dia due to slumping.

† W pellet interior after edge ridge worn away — Ridge — 0.2

? probably slightly less due to exclusion of ridge volume and linear ave ~~approx~~

201-10 after flattening; 15 μm

0.90† ~1.057 0.168 0.147 6.12 85 better (more accurate)

" 2.96 (2.98)

201-11 1.357 0.412 0.596 4. ~~4.96~~ 5.0 (69.4)

5/12 2.935 ~1.315 0.365 0.496 5.94 82.5

2.945

201-11 cut "in half" larger flattened and polished.

0011-2201 sandwich ~0.353-0.363 thick.

> from furnace top to bottom of "weight plate" 1 9/32" @ 462C
assuming ~6 lbs for ram & plate & x-sectional pellet area
of 0.212 in² load ⟹ 28 psi

T diffusion sintering set @ 860C for ~12 hrs.

Rel density from measurement of 201-11 ~83 %. On inspection of internal polished surface numerous burnout-like occlusions present. Some degree of open porosity, also.

Pyc. rel. den = 88. % thus Δ attributable to open porosity.

0011 rel density from measure ~ 89 %. No pyc reading done. 16h sinter @ 975C.

4:30 pm T @ 859C assume start of diffusion sintering

Plate height 1 3/8" (3/32" expansion due to TCE from 462C & No RT measure made, but not significant)

11-28-88 <INSERT>

Results (by microprobe) of CaSrCuOx melt xstals

Melt composition was from pgs. 27-29

Composition was <u>not</u> $Ca_{0.86}, Sr_{0.14} Cu_1 O_x$ in melt, but rather

→ $Ca_{0.48} Sr_{0.14} Cu_1 O_x$ ←

from which xstals grew of CaSrCuOx with stoic.

$Ca_{0.81} Sr_{0.2} Cu_1 O_x$ $x = 2.1$

Atomic wgt fractions were:

Ca	0.195
Sr	0.05
Cu	0.242
O	0.513 (by difference)

Melt temp. was 1000C for 16h with cooling virtually but NOT TOTALLY A QUENCH: UNCONTROLLED RATE REGULATED BY FURNACE thermal MASS

Date and sign every entry. Hav... possibly important entry witnessed. Submit an In... ...closure of anything possibly new and inv...

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11-22

Balance Bi Powders for Rxn

~g

2212 - 30.5

2201 - 12.5

0011 - 33.5

2nd Diffusion Run 2hr ramp to 866C @ 100C plate space = 1 7/32

0011 slice started @ 0.18 cm (not measured when finished, either was 2201 or sandwich, will try to approx)

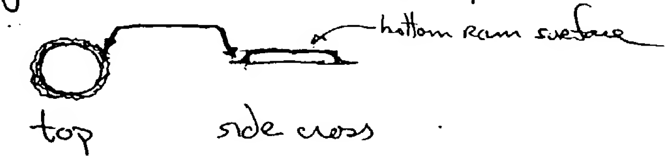
2201 ~ same 0.17

loose ~ 0.23 - 0.2 ≃ 0.03/slice ∴ 0.18 → 0.15, 0.17 → 0.14
so sandwich might be ~ 0.29 cm (80% of run #1)

Approx thickness by height of plate differences @ 866C 1 12/32 - 1 9/32

3/32 = 0.23 cm ∴ ~~.29~~ - 0.23 = 0.06 too small → 871C peak

RESULTS: "Bi" pellet has spread, apparently melting. Total thickness 0.18 cm.
Liq ~~...~~ generated "crystalline" (?) skirt around pellet periphery.



0.18 cm = 0.07" slice ~ square 0.07 + 0.015 = 0.085

The above understood and witnessed by ______ Date ______ and by ______ Date ______

0011 - 2201 Mix Calculations

wt % 0011	wt % 2201
~~2~~ ✓	+2
~~8~~ ✓	+5

$Bi_2 Sr_2 Ca_1 Cu_2 O_8$
$Bi_2 Sr_2 Ca_0 Cu_1 O_6$
$Bi_0 Sr_0 Ca_1 Cu_1 O_2$

From "ideal" stoic. 2201 + 0011 → 2212
1 mole 1 mole 1 mole

	A.W.	0011	2201	2212
Bi	208.98	—	417.96	417.96
Sr	87.62	—	175.24	175.24
Ca	40.08	40.08	—	40.08
Cu	63.54	63.54	63.54	127.08
O	15.9994		95.9964	127.9952

135.6188 + 752.7364 = 888.3552

	0011	2201	2212
	0(0.14)(0.86)1	(2.15)(1.6)(0)(1)	(2.15)(1.68)(1.17)(2)
Bi	—	449.307	449.307
Sr	12.2668	140.192	147.2016
Ca	34.4688	—	40.328496
Cu	63.54	63.54	127.08
O	31.9988	~95.9964*	~127.9952
	142.2744	749.0354	891.912296 / (891.3098)
% dev	+8.6%		

99.93%

*(5.825)15.9994
93.19651

99.6% (2.15 etc)

CONTINUATION...

1 mole "0011" + 1 mole 2201 ~~⇌~~ 011 + 529 wt % 2201

142.2744g   752.7364g

142.2744g + (0.02)(142.2744) = 145.12 g
2.84549
factor 15   9.49 ~~9.485~~   0.19 = 9.675   2 wt % batch size

142.2744g + (0.05)(142.2744) = 149.~~38812~~39 g
7.11372
* 9.485   ~0.~~475~~ 0.48 = 9.96   5 wt % batch size

For Stoic (molar) mix = 1.~~423~~143 g + ~~7.527~~ 7.53 = 8.95g batch size

Total Usage   0011   2201   g

20.393   8.192   g

%   61   66

	cc	cc	vol %	wt %
Vol table	2.37	0.0264	1.0%	2
	2.37	0.0736	3	5
	0.366	1.045	25%	stoic

STOIC:	Bi	Sr	Ca	Cu
"2201"	2.15	1.6	0	1
"0011"	0	0.14	0.86	1
	2.15	1.74	0.86	2

versus poly 2.15 1.68 1.17 2

 Stoic Mixing

0011 - 2201

~1.43g ~7.53 MIX STARTING @ 3:00 P.M., 50mls isopropyl.
5cc ZrO_2 balls
2/3 full

NOTE: from bottom pg 51 can be seen this additive approach will yield a theoretical molar comp { 0.1 M larger in Sr / 0.31M less in Ca

i.e. ~~Strontium rich~~, Calcium poor

8.96g added initially, 8.85g recovered ∴ 1.2% loss (98.8 yield)

Stoic 1 Pre 2700/27,500

3.11 1.36 0.486 0.706 4.41 ~68.9

0.25 (4) + 0.75 (7.2) = 6.4 vol % basis, ~ density calc

Rxn. (SINTER) temp to be 850C

Pellet melted indicating lower mp liq ϕ exists in system & later xstallized. Predominantly 1 lath-like ϕ w/ exaggerated growth as in 2201 120h sample.

12-5

4:20 P.M. // 4:25 @ temp

0011-3 placed in preheated Rapid temp set @ 951C (T_{samp} = 975C) for overnite sintering.
No pre data on density due to irregular shape caused by pellet crumbling during isopressing.
unipress → 6000 , 150 - 29,000 PSI wght ~ ~~3.1~~ 3.0-2.9 g

12/6 9:30 Slow cooling begun ∴ ΔT_{sinter} = 17h @ 875C

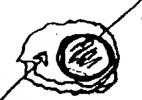
Post 2.86g ~0.460 mm thick radius might have been ~1.38

estimated density 0.666cc @ 3.1g ~ 4.6/5.00 ≅ 93 (may be high)
3.0 4.5 / ↓ 90 better

0011-3
0.181" thick
Slice 1 → 0.09" after cleaning // post polish → N/R
Slice 2 → 0.074
0.179

2201-8 1.038 dia ∴ area = $\pi D^2/4$ = 0.85 cc^2
0.409 = 0.525 in^2 5.75 lbs/.525 in^2 ~ 11 psi

2201-8 (top)



0011-3

Pellet configuration @ START ~ 3:55 p.m. thickness - 0.34 cm

Ramp → 434 Set point - 800C Dwell - 12h 1 7/32 @ 380C

12/7 Result: No melting, pellets bonded w/ little deformation.

12/8 After 24h 825C anneal no evidence of liq., but bond breaks after handling at pellet interface with some "RXN etching" of 0011 pellet surface leaving thin, uneven layer of 2201 (or RXN prod) behind.

12-6

IBM Technical Notebook

~~Ref pg 25~~

SECOND 2201 Synthesis $Bi_{2.15}Sr_{1.6}\cancel{Ca}Cu_1O_6$

Bi as Bi_2O_3 : 30.0543 x 2 = 60.1086 . 60.11

Sr as $SrCO_3$: 14.1724 28.3448 28.35

Cu as CuO : 4.7724 9.5448 → 9.54

48.9991 g 97.9982 g

~ 0.7019 conversion factor for $CO_3 \to O$ 28.3448 (0.702) = 19.898

Estimate ~ 89 g "batch recovery" 97.9982 − 8.4468 CO_2 loss = 89.55

12-7

tare 202.54
Bi_2O_3 262.68
60.13 − 60.11 = +0.02 ✓

~~tare 262.~~
$SrCO_3$ 291.03 ← 291.28.35 wgd 28.36 transferred 28.36
tare 262.68
28.35 Δ ✓✓

CuO 300.57 (300.57/9.54) wgd 9.55
291.03
9.54 Δ ✓

12-8 97.92 / recovery after drying overnite
98.02 theoretical = 99.9% yield 0.1% mixing loss

to pg 56 →

12-7-88

011	2201	
9.49	0.48	theor.
9.48	0.50	wgd
0.02	>.01	Read
9.46	0.49	actual = 9.95

Std. 1-1.5h 5min ZrO_2/Iso grind mix, screening & drying.

12-8-88

Recovery : 9.84g / 9.86g theoretical = 99.8% > 0.2% loss

60.87
cont tare 51.04/5
9.83 transferred

0011-2201-5W(3V)-1
Post 8500/29,000
2.31 1.117 0.704 0.690 3.35 ~67%

Pellet bigger than usual, 1.75g max in future might be considered.

— 1.183 0.715 cracked, measurements N/A

12-9
5W-Z 900C 8500/30,000

1.27 1.174 0.382 0.414 3.07 61.4!

3:55 in preheated furnace → 4:00 to temp @ 900C
POST 5 MIN
1.24 1.111 0.36 0.349 3.55 71 —

15 MIN NO SIGNIFICANT CHANGE

12-12 to temp ~ 10:30 A.M. (check: 10:45 → no slumping) → SINTER till 12:30
12:15 A.M slow-cool initiated
~~11:45~~.
~2h
1.24 1.055 0.33 0.29 4.28 ~86%

12-8-88 2201 SYN II cont. (from pg 54)

186.68 → spill → 186.69 → 192.29 w/ top
crucible tare 88.79
97.89

10:00 A.M → 575C hold 1h
11:00 → 800C
12-9 11:00 AM cool, reground to < 100 mesh

182.23
88.95 (wgt after sintered powder body removed)
93.28

~~93.44~~ if 88.79 used
181.02 after grinding
88.95
92.07 to temp. (866 C.) @ 1:00 p.m.
−Δ 1.21 in grinding 1.3%
97.89 − 93.28 = 4.61
97.89 − 89.55 = 8.34
55% REACTED

1:00 − 5:00 pm 866C, shut down for weekend (away restart sun eve)

12-12-88

to temp 866C @ 10:00 A.M.
off @ 7:00 A.M. 12/13/88

PARTIAL MELTING, "classic" EUTECTIC lamillar amid large 2201 lathes.

~~12-14-88 2201 Syn III~~

~~mixing jar time: 202.75~~

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Date and sig... ...ry entry. Have every possibly important entry ... ...submit an Invention Disclosure of anyth... ...bly new and inventive.

 12-~~14~~15-88 SynIII 2201 IBM Technical Notebook

$Sr/Bi = 0.744$

Sr 0.8 Sr = 1.72

mixing jar tare 202.75

x 2

Bi as Bi_2O_3	30.0543	=	60.11
Sr as $SrCO_3$	14.1724		28.35
Cu as CuO	4.7724		9.55
			98.01
			− 8.5 CO_2 loss
			89.51

Bi_2O_3 262.86
tare 202.75
60.11 ✓
291.20(19)
262.86
28.34(5) ✓
300.75
291.20
9.55 ✓

PRE CAL I

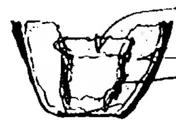
crucible+ 185.84
tare 87.99
97.85 97.85/98.01 0.2% mix loss

12-20 Post 750C 16h calcination

crucible+ 181.15 NO RXN w/ Pt.; lime green cute/bottom, uniform throut
87.98 except for top ½ edges (grey)
93.17 93.17−97.85 = −4.68 /8.5 = 55.% CO_2 lost
post grind 92.70 post grind

crucible+ 180.70
87.98
(92.72)

12-21-88 Post 790C 20h calcination
178.10 Material looks very good, smells indicating lamilar structure
87.98 Uniformly black, sparkling, sinter body. An outer shell
90.12 / ~89 ≃ RXN close to completion ≈ 98+%
and inner core structure, (see below)



to page 60

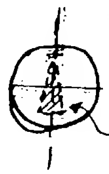
The above understood and witnessed by ______ Date ______ and by ______ Date ______

12-14-8

$Y_1Ba_2Cu_3O_x$ Implantation Experiment

PRE - film on $SrTiO_3$ 3500/30,000

3.07 ~~0~~.448 0.485 0.799 3.84 ~60.4 %

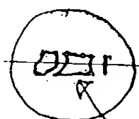


← line 'marker' ⊥ to long axis of triangular $SrTiO_3$ implant

implant orientation - NOTE: MARK ON UNDERSIDE of pellet ∴ film side opposite

3.02 1.271 0.396 0.50~~2~~ 6.02 94-95

3.05 1.448 0.476 0.784 3.89 ~61



← line 'marker' ∥ to long cutting axis of two pellets (cut on line

implant orientation - see NOTE above for polished side orient.

2.99 1.272 0.391 0.497 6.02 94-95

5

5:12 p.m. 475C @ 10C/min to 975 Δ500C/10C/min = 50 min ~ 6:00 pm.

Cutting $SrTiO_3$ implant: measures 0.5" on saw (0.0~~2~~5-0.505 tangents)

$\frac{1.272}{2.54}$ = 0.48~~05~~ ✓

12-21-88 Calcination III 2201-B3

X-RAY SHOWS DISTINCTLY NOT SINGLE ϕ, even though material looks "O.K."

total 177.04
cruc 87.98
89.06 88.2

- 87.98
+ 88.2
0.22 g GAIN

12-22-88
175.7– (–Δ 1.34) slight sticking (RXN) w/ cruc. bottom
87.98
87.72 – 89.51 (theo.) = 1.79 g greater than theoretical loss
could be grinding loss 2%

PRE 850 cal
174.93 total
87.93 tare (after acid cleaning)
87.– g 0.72 g grinding loss (consistent w/ previous losses) + x-ray slide

POST 12-22-88
174.83
87.93
86.90 – –Δ 0.10 100% RXN ! ~ constant weight

NOT superconducting but not surprising batch #1 wasn't either.

Summary various RXN pellets:

5 wt% 2201 in 0011 for 16h @ 850C SEM
5 wt% 2201 in 0011 for 2h @ 975C SEM
[0011 - 2201 STOICH MIXTURE ~~pressure bonded~~ pellet: 13h 850C
liq ø formation, exagerated grain growth/warpage] later
0011 @ 975C 17h SEM STD.
2201 @ 875C 1h SEM STD.
2212 @ 853C 5 MIN SEM STD.

12-29-88 Dave's Compositions

#	Y	Ba	Cu	Y	Ba	Cu
	1 (0.167)	2 (0.33)	3 (0.50)	0.17	0.33	0.50
	0.15	0.33	0.52	0.8654	1.9038	3 —
	0.17	0.35	0.48	1.0625	2.1875	3 —
	0.19	0.33	0.48	1.1875	2.0625	3 —
	0.19	0.31	0.50	1.14	1.86	3 —

Calculated Compositions (calculations next page)

#	Y	Ba*	Cu	total
1)	1.91937 (1.92)	6.51253 (6.51)	3.97697 (3.98)	12.48
2)	1.69356 (1.69)	6.51253 (6.51)	4.13605 (4.14)	12.34
3)	1.92	6.90723 (6.91)	3.81789 (3.82)	12.65
4)	2.14578 (2.15)	6.51 0.33	3.82 0.48	12.48
5)	2.15	6.1783 (6.18) .31	3.98	12.31

* Ba as $BaCO_3$
Y as Y_2O_3
Cu as CuO

} NOTE ⇒ No purity corrections applied yet

1/3/89 Calculations for weights SUMMARIZED ON page 62

2) $Y_{0.15}\ Ba_{0.33}\ Cu_{0.52}$

$Y = 0.15\,(225.8082)/2 = 16.9356$ g Y_2O_3

$Ba = 0.33\,(197.3494) = 65.1253$ g $BaCO_3$

$CuO = 0.52\,(79.5394) = 41.3605$ g CuO

3) $Y_{0.17}\ Ba_{0.35}\ Cu_{0.48}$

$Y = 0.17\,(225.8082)/2 = 19.1937$ g Y_2O_3

$Ba = 0.35\,(\quad)$ 69.0723 g $BaCO_3$

$Cu = 0.48\,(\quad) = 38.1789$ g CuO

4) $Y_{0.19}\ Ba_{0.33}\ Cu_{0.48}$

$Y = 21.4518 \quad Ba = 65.1253 \quad Cu = 38.1789$

5) $Y_{0.19}\ Ba_{0.31}\ Cu_{0.50}$

$Y = 21.4518 \quad Ba = 61.1783 \quad Cu = 39.7697$

1) $Y_{0.17}\ Ba_{0.33}\ Cu_{0.50}$

19.1937 65.1253 39.7697

1/3/89

 ~~Ag~~ (TRIAL #1) **IBM Technical Notebook**

1) O_2 FIRED ↳ 88.5409 $Y_{.17}\ Ba_{.33}\ Cu_{.5}$

Y_2O_3 - 1.92   CuO - 3.98(1) $BaCO_3$ - 6.51 / 6.57~~88~~ (77) 6.5883

88.53	2.31~~40~~ ✓	4.3712	6.9665 / ~~6.9785~~ 6.9685
tare 76.06	0.3900	0.3900	0.38~~88~~
12.47	1.922	3.98(12)	6.5777 = 12.48
post dry 12.43			

↳ 99.6% recovery total - Δ = 0.05

1-9-89

10.95 g after 2nd 16h 950C O_2 calcination

$6.5777\left(\frac{153.3394}{197.3510}\right) = 5.11$ $-\Delta$ 1.47   12.43 − 1.47 = 10.96 ✓

0.777

1/17 D(AVE) post 1 h grind = 1.86 μm   3000/30,000

P1   1.68   1.136   0.408   0.4135   4.06   63.8%

16h In O_2 @ ~3:00 p.m. 1/10/89, to temp @ 950C projected ~4:30 → 5:00 → 9:00

1.64   1.011   0.354   0.284   5.775   90.8 (91)

The above understood and witnessed by   Date   and   Date

1/4/88

4) ~~BR~~ fired $Y_{0.19}$ $Ba_{0.33}$ $Cu_{0.48}$
O_2

~~1.89~~ 6.51 ~~4.14~~ 3.82
~ 2.15

3.8217
.3865
4.2082

Y_2O_3 - 2.5342 6.9623 4.2083
0.3865 0.384 0.3865
2.1477 6.5783 3.8218 ⇒ 12.55 g

2.1473
.384
2.5313

1/10/88
Second ~~cal~~ CALCINATION STARTED after grinding. No evidence of liq formation. Powder looks good already.

10.99 g after 2nd calcination;

10.76 post grind

~~12~~ 6.58 (.777) = 5.11 → Δ 1.47

12.55
1.47
11.08 g expected: 10.99 Recovered

1/17
P1 PRE 2500/30,000 to temp @ ~ 5:00 p.m.

1.60 1.14 0.399 0.4073 3.93 62 %

1/18
1.58 1.055 0.365 0.319 4.95 77.8 %

Green ϕ peaks coming up in X-ray.

5) $Y_{0.19}\ Ba_{0.31}\ Cu_{0.50}$

(.999) Y_2O_3 - 2.14(73) 2.3760 target $BaCO_3$: 6.17(96) ① /.99 ⇒ 6.2407   CuO- 3.98(10) ② 4.2092 target

2.3761 / 0.2287 / 2.1474 ✓   tare 6.4690 / 0.2229 / 6.2411 6.4686 target   4.2092 / 0.2282 / 3.9810 ⇒ 12.3695 ~12.37

12.34(3) collected after mix // 12.34/12.37 ~ Δ 0.24 % ✓

6.2411 - 4.8493 = 1.392

tare 63.50 / 51.17 / 12.33   62.20 / 51.17 / 11.03   -Δ 1.3/1.39 = 93.5%

11.00 POST GRIND   62.17 / 51.18 / 10.99   POST CAL II 62.11   11.63 / -.88 / 10.75

Post grind 10.46
P1 3333/30,000 ← PRE →
1.60 1.440 0.244 0.40 4.00 62.9 ✓
Post 1.56 1.266 0.210 0.26(?) 6.00 (94 ⇒ 91)

Good densification, no apparent liq, CuO islands present.

Date and sign every entry. Hav... entry witnessed. Submit a... Inve... anything possibly new a... ...en... ...ry possibly important Disclosure of

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2201

P1B3 2500/30000
PRE
1.94 1.09 0.391 0.365 5.315 73.8%
POST 15 min @ 860
1.94 1.06 0.35~~9~~ 0.313 6.2 86%

2 wt% 2201 in 0011 1/13/88

9.49
0.19
9.68

some leaking during 0.5 h mix : 9.05 g

9.05 g
8.68 g final
0.37 g loss

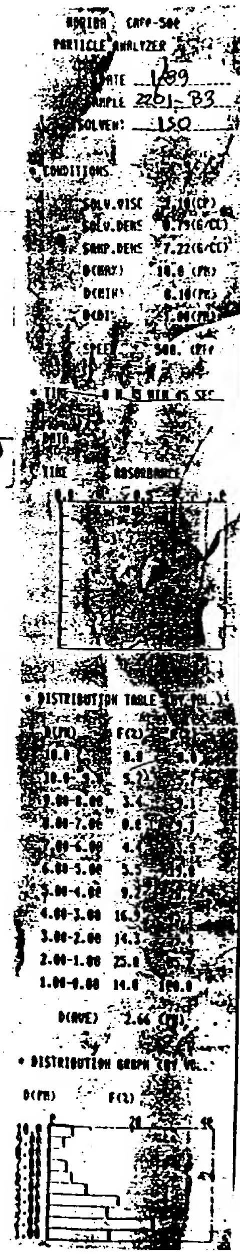
8.68 g
0.37 g

2 wt% P1 4200/30,000 to temp @ ~5:00 p.m.
Pre
1.70 1.163 0.499 0.53 3.21 ~64.2 ~~(80.25%)~~

1.69 ~1.21 0.525 0.60 2.82 56.4
4
1.191 - 1.227
some slumping -Δ 12%

Cuts yield : 1.57 mm inside ~0.30 cts
1.12 mm center 0.25
polished → 1.79 mm outside 0.380 (tan)

\+ 26.52 26.03 25.94
post 25.52 25.52 25.45
1.00 → 1000 μms 510 490



The above understood and witnessed by Date and Date

2) $Y_{0.15}\ Ba_{0.33}\ Cu_{0.52}$ bottle tare: 74.55

Y_2O_3 - 1.6936 ~~BaO~~-6.5125 CuO- 4.1361.
$BaCO_3$- 6.5783

(.22705) 1.9206 / 0.2270 / ~ 1.6936 6.8066 / 0.2284(8) / 6.5782 - 4.3651 / 0.2288 / 4.1363 - (0.0084) ~12.41 total

1/17, 1ST CALCINATION tare 66.53 / 54.16 / 12.37 (12.37 measured from mixing) ✓/12.41 = -Δ 0.3%

6.5783(.222) = 5.111 (-1.47) 12.37 / - 1.47 / ~ 10.90 expected yield (less transfer losses)

1/18 post 66.53 / 65.15 (05) / -Δ 1.38 (48) tare 54.20 recovery 65.05 / 54.2 / 10.85 ~total RXN.

2nd CAL (16h as above)

1/19 POST 65.02 / 54.2 / 10.82 ✓✓ ~ constant 10.79 recovery

Notes: large liq. stains (formation) during 1st/2nd cal unlike 1 & 4 where liq was suppressed in 1st cal & minor in 2nd.

PRE P1 3300/30,000 75C @ 5:16 o. temp @ 7:45, 16h → 11:45 AM
6:40 7:45

1.60	1.414	0.258	0.405	3.95	62.1%
1.57	1.227	0.216	0.255	6.16	96.9

1h iso PSD's

1) O_2 4) O_2 2) O_2 3) O_2 5) O_2

1) O_2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1-10-89
SAMPLE YBaCuO
SOLVENT ISO-1h

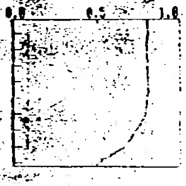
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

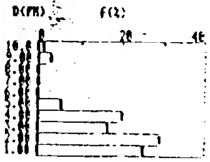


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.2	1.2
9.00-8.00	2.9	4.1
8.00-7.00	0.0	4.1
7.00-6.00	0.0	4.1
6.00-5.00	0.0	4.1
5.00-4.00	5.5	9.6
4.00-3.00	19.9	29.5
3.00-2.00	16.5	46.0
2.00-1.00	28.9	74.9
1.00-0.00	25.1	100.0

D(AVE) 1.80 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



4) O_2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1-13-88
SAMPLE YBaCuO
SOLVENT ISO

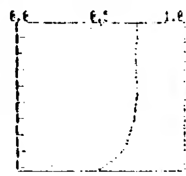
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.7

TIME ABSORBANCE

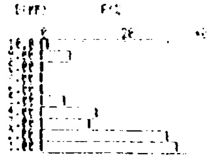


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.2	1.2
9.00-8.00	6.9	8.1
8.00-7.00	0.0	8.1
7.00-6.00	0.0	8.1
6.00-5.00	0.0	8.1
5.00-4.00	5.2	13.3
4.00-3.00	13.2	26.5
3.00-2.00	11.4	38.0
2.00-1.00	29.8	67.8
1.00-0.00	32.2	100.0

D(AVE) 1.60 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



2) O_2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1/20/89
SAMPLE YBaCuO
SOLVENT ISO

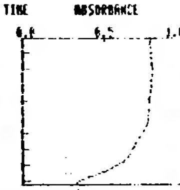
• CONDITIONS D- 0.8

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

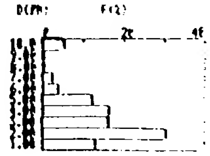


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	5.4	5.4
9.00-8.00	0.0	5.4
8.00-7.00	0.0	5.4
7.00-6.00	2.3	7.7
6.00-5.00	3.8	11.4
5.00-4.00	12.2	23.6
4.00-3.00	16.2	40.0
3.00-2.00	16.3	56.3
2.00-1.00	30.0	86.5
1.00-0.00	13.1	100.0

D(AVE) 2.39 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



3) O_2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1/27/89
SAMPLE YBaCuO
SOLVENT ISO

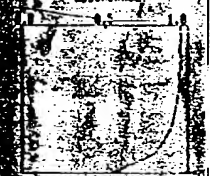
• CONDITIONS D- .96

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE



• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	1.0	1.0
8.00-7.00	3.5	4.5
7.00-6.00	4.1	8.6
6.00-5.00	[illegible]	15.0
5.00-4.00	7.7	22.7
4.00-3.00	11.8	34.5
3.00-2.00	14.3	[illegible]
2.00-1.00	28.0	[illegible]
1.00-0.00	23.2	[illegible]

D(AVE) [illegible] (PM)

• DISTRIBUTION GRAPH (BY VOL.)

5) O_2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1-24-89
SAMPLE YBaCuO
SOLVENT ISO

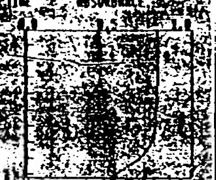
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	[illegible] (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE



• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	[illegible]	[illegible]
10.0- 9.0	0.0	[illegible]
9.00-8.00	<0.0	[illegible]
8.00-7.00	[illegible]	[illegible]
7.00-6.00	[illegible]	[illegible]
6.00-5.00	[illegible]	[illegible]
5.00-4.00	[illegible]	[illegible]
4.00-3.00	[illegible]	[illegible]
3.00-2.00	21.6	[illegible]
2.00-1.00	26.6	[illegible]
1.00-0.00	34.5	100.0

D(AVE) 1.50 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

		SrO	CuO
$Sr_7Cu_{12}O$	$\rightarrow Sr_{0.37}Cu_{0.63}O_{19}$	38.34	50.1078
$SrCuO$	$\rightarrow Sr_{0.5}Cu_{0.5}O_x$	51.8097	39.7697
Sr_2CuO	$\rightarrow Sr_{0.67}Cu_{0.33}O_3$	69.4250	26.248

$SrO = 103.6194 \rightarrow SrCO_3$ 147.63 14247

$CuO = 79.5394$

3.83 5.02

3.834 5.0178 → 8.8538

(5.18) 5.18097 3.97697 (3.98) → 9.1579

(6.94) 6.9425 26.248 (2.63) → 9.5673

5.46	5.02	10.48
7.38	3.98	11.36
9.89	26.34	12.52
SrCO	CuO	

1/17/89

CO11-5 1% 201 @ 850C for attempted TEM prep.

slice 2 - 1.28 mm - 1280 μm

slice 3 - 0.68 680 μm

slice 2 prep: mounted side 1 measures ~ 27.64
1280 26.30-(33)
~ 1.34 - 1.29

aim: 300 μ 1340
980/2 = 490 - 490
850 target

∴ 20 8's on "soft" 15 μm grits ~ 900 ; 150 8's on 6 give 770

720 μm before starting second side

26.40 after mount ~~30~~ → 26.21 ~~150~~ → 26.16 150 6's 26.06
25.69 25.69
0.71 ✓ 520 ~430 330 ✓

3) O_2 fired

$Y_{0.17}Ba_{0.35}Cu_{0.48}$

Y_2O_3: 1.9213 $BaCO_3$: 6.9770 CuO: 3.8217

2.1496 7.2047 4.0498

2.1495	7.2050	4.0498
0.2283	tare 0.2277	0.2281
1.9212	6.9773	3.8218
	5.4214	
	1.55	

-Δ 3.%

Some bumping, but very good mix so should be fine. 12.35 post mix

12.72 g expected REDO (Bumping too critical)

Y_2O_3: 1.9213 $BaCO_3$: 6.9770 CuO: 3.8217

2.1425 2.1413	7.1973 1.1972	4.0428	4.0427 total
0.2210	0.2200	0.2210	
1.9215	6.9773	3.8218	12.72

Mix recovery after overnite drying: 12.63/12.72 -Δ 0.7% exceptable

~ 63.82
tare 51.19

Post 62.39
51.20
11.19

(11.17 expected)

CAL #I: 3:57 190C ~5:15 est. to temp attainment
4:25 486C ~ ✓ seems correct

Post cal II
62.24
51.20
11.05

Post grind I: 11.21 → 11.14 62.35

Very little liq formation compared to #2. Apparently need excess Ba and Cu for large liq..

0.2 g loss due to Si contam from tube (pre-grind) 10.8

1/27

10.97 collected: white top layer on powder. Dry flake-cake very agglomerated/brittle and does not easily powder out when brushed. Need to dry grind in order to produce decent powder.

10.25 recovered after dry grind

P1 3500/29000

1.62	1.425	0.266	0.424	3.82	60 — %
1.52	1.321	0.245	0.336	4.52	71 %

Pre grind & Post grind x-rays show change of some peaks in two x-rays, however sintering calcination of pellet may return rxn products to original ϕ's. Will do x-ray of pellet also.

Post: some slumping.

 2/16/89 123 Variation Study IBM Technical Notebook Property Pellets

#1 P2 3000/50,000 ave 62.5

1.04	1.138	0.256	0.260	4	62.9
to 1.01	1.046	.22	0.189	5.34	84

#2 P2

1.02	1.123	0.264	0.261 (5)	3.90	67.5
1.00	.981	.223	0.168 (6)	5.95	93.6

#5 P2

1.04	1.154	0.248	0.258	4.03	63.4
1.024	1.024	.224	0.184	5.54	87

Pellets in furnace @ ~10:30 A.M. 2/17/89 10°/min (pre-warmed)

t	T_c	T_s	
10:30	259	239	(?)
10:45	442	518	+Δ 75 Ts
11:50	900 sp	944	

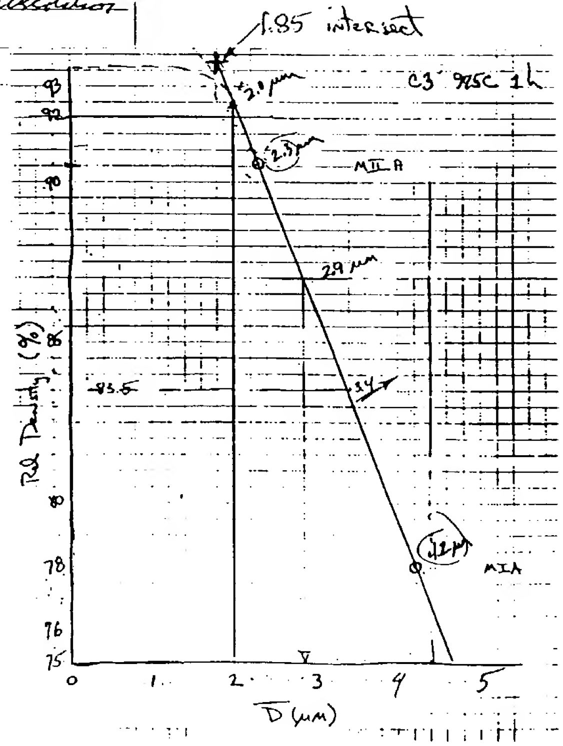
from early results Δ @ 800 went down by 25 so estimated time to reach set point would be 45 mins. or 11:30 @ +Δ50 overshoot or 950 C then relaxation over ensuing 20 mins of 6°C. Relay in pellets run suggests the time was 13C so probably close.

1:45 pm 2/17/89 in furnace @ 600C project out 72 hrs.) 1:45 Mon.

See page 76

Experiments to look at.
Carbonate in 123
Clean? gas with Ascarite
Sample 1 dense closed porosity, $\rho \geq 91\%$
cut sections from center

Sample 2 open porosity
$\rho < 87\%$
center section
thin slices oxygenate
(3) 3 pellets of each (Peter)
center sections of each
(1) TEM/EELSS (P. Batson)
(2) Magnetometer (T. Moquino)
(3) Induction (Diane Dimos)
(4) CO_2 evolution on dissolution
(5) X ray lattice
( ) XPS



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/7/89
SAMPLE C3 PA 5b
SOLVENT ISO

• CONDITIONS MIIA

SOLV.VISC [illegible]10(CP)
SOLV.DENS [illegible]79(G/CC)
SAMP.DENS [illegible]36(G/CC)
D(MAX) [illegible]0 (μm)
D(MIN) [illegible]10(μm)
D(DIV) [illegible]00(μm)

SPEED [illegible]00. (RPM)

• TIME [illegible] MIN 20 SEC

• DATA 0.8

TIME ABSORBANCE

• DISTRIBUTION TABLE (BY VOL.)

D(μm)	F(%)	R(%)
[illegible]	[illegible]	0.0
[illegible]	[illegible]	0.0
[illegible]	0.0	0.0
[illegible]	0.5	0.5
[illegible]	3.7	4.2
[illegible]	[illegible]	9.4
[illegible]	[illegible]	16.8
[illegible]	14.5	31.3
[illegible]	28.3	59.6
2.00-1.00	30.1	89.7
1.00-0.00	10.3	100.0

D(AVE) 2.34 (μm)

• DISTRIBUTION GRAPH (BY VOL.)

D(μm) F(%)

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/7/89
SAMPLE C3 PI [illegible]
SOLVENT ISO

• CONDITIONS [illegible]I

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 0.0 (μm)
D(MIN) 0.10(μm)
D(DIV) 1.00(μm)

SPEED [illegible]00. (RPM)

• TIME [illegible] MIN 20 SEC

• DATA ~0.8

TIME ABSORBANCE

• DISTRIBUTION TABLE (BY VOL.)

D(μm)	F(%)	R(%)
10.0<	[illegible]	0.0
10.0-9.0	[illegible]	3.6
[illegible]-8.00	[illegible]	12.1
[illegible]-7.00	[illegible]	22.[illegible]
[illegible]-6.00	[illegible]	27.[illegible]
[illegible]-5.00	[illegible]	39.[illegible]
[illegible]-4.00	[illegible]	52.0
[illegible]-3.00	[illegible]	64.7
[illegible]-2.00	[illegible]	78.2
[illegible]-1.00	[illegible]	92.9
[illegible]-0.00	[illegible]	100.[illegible]

D(AVE) [illegible] (μm)

• DISTRIBUTION GRAPH (BY VOL.)

D(μm) F(%)

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/8/89
SAMPLE C3 5b
SOLVENT ISO

• CONDITIONS MIVA

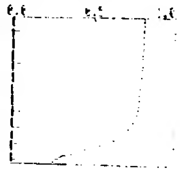
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (μm)
D(MIN) 0.10(μm)
D(DIV) 1.00(μm)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.8

TIME ABSORBANCE



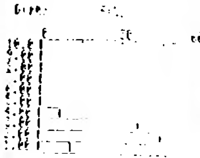
• DISTRIBUTION TABLE (BY VOL.)

D(μm)	F(%)	R(%)
10.0<	0.0	0.0
10.0-9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	0.0	0.0
6.00-5.00	0.0	0.0
5.00-4.00	0.0	0.0
4.00-3.00	[illegible]	7.3
3.00-2.00	[illegible]	43.5
2.00-1.00	44.[illegible]	88.[illegible]
1.00-0.00	[illegible]	100.0

D(AVE) 1.85 (μm) ✓

• DISTRIBUTION GRAPH (BY VOL.)

D(μm) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/8/89
SAMPLE C3 5b
SOLVENT ISO

• CONDITIONS MIII A

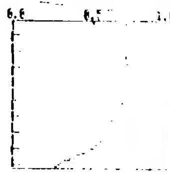
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (μm)
D(MIN) 0.10(μm)
D(DIV) 1.00(μm)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.8

TIME ABSORBANCE



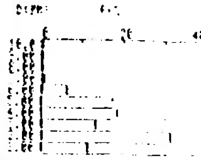
• DISTRIBUTION TABLE (BY VOL.)

D(μm)	F(%)	R(%)
10.0<	0.0	0.0
10.0-9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	0.5	0.5
6.00-5.00	6.4	6.9
5.00-4.00	17.7	24.5
4.00-3.00	18.7	43.2
3.00-2.00	13.5	56.6
2.00-1.00	31.8	88.4
1.00-0.00	11.6	100.0

D(AVE) 2.56 (μm)

• DISTRIBUTION GRAPH (BY VOL.)

D(μm) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 2/19/88
SAMPLE C3 5b - Sims
SOLVENT ISO

• CONDITIONS

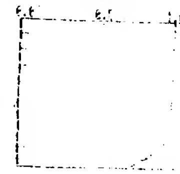
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (μm)
D(MIN) 0.10(μm)
D(DIV) 1.00(μm)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.93

TIME ABSORBANCE



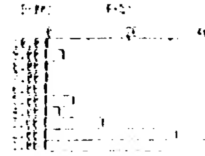
• DISTRIBUTION TABLE (BY VOL.)

D(μm)	F(%)	R(%)
10.0<	0.0	0.0
10.0-9.0	0.0	0.0
9.00-8.00	4.2	4.2
8.00-7.00	0.0	4.2
7.00-6.00	0.0	4.2
6.00-5.00	0.[illegible]	4.4
5.00-4.00	6.8	11.[illegible]
4.00-3.00	3.[illegible]	14.3
3.00-2.00	13.9	28.[illegible]
2.00-1.00	31.8	60.[illegible]
1.00-0.00	35.5	100.0

D(AVE) 1.32 (μm)

• DISTRIBUTION GRAPH (BY VOL.)

D(μm) F(%)



84

94-95
2/8/[illegible]

95
[illegible]

The above understood and witnessed by
Date
and
Date

2/7/89

ρ < 87% PI mill $\bar{D}$ = 4.21 μm (quick)
3333/28500
Pre wght: 3.46 (of 3.5) some ~~pounding~~ deformation during isopressing, but Δ wght minimal so ~ density can be deduced.
Post 975C 1h

3.41	1.402	0.448	0.69	4.94	77.7 → 78%	4.2 too low

PII mill $\bar{D}$ = 2.34 μm (slow)
Pre 3900/29,000

3.46	1.453	0.543	0.9	3.84	60.5	~ reasonable
3.40	1.285	0.456	0.59	5.76	90.6	<NEED SLIGHTLY HIGHER>

Tomorrow → will mill un

~~Pre~~

Yields: Pre ~~50~~ MI : Post 48g — grp B
grp A { 24 MIIA: 20g — 20.5
grp A { 16 MIIIA: ~~4~~

error in pre-weights

78 M4A | $\bar{D}$ = 1.85 PRE - 4000/30,000 [20°/min to 675, 10°/min to 975; 1 h 975C { quench]

P3	3.40 ~~3.48~~	1.452	.543	0.9	3.78	59.4	
	3.48	1.265	0.460	0.578	6.02	94.6	vent
P4	3.41	1.462	.540	0.91	3.75	58.–	
	3.60	1.274	0.468	0.596	6.04	95–	hairy
P5	*3.43+	1.457	0.561	0.935	3.67	57.7+ → ≥ 58	
	3.60	1.280	0.482	0.62	5.81	91.4+	{ Probably even higher
	3.65				5.89	~~92.6~~	

MIIB - 4000/30,000 30 μm

P6	3.55	1.463	0.508	0.85	4.17	65.6	
	3.53	1.36	0.464	0.674	5.24	82.4	vent
P7	3.54	1.462	0.504	0.85	4.16(5)	65.5	
	3.59	1.356	0.462	0.668	5.36	84.5	hairy
P8	3.56	1.463	0.507	0.85	4.19	65.9	
	3.73	1.352	0.453	0.65	5.73	90.2	

* laminated on 1 side, not severe (MUST BE 'repelletized' pellet)

6:15

P9 4000/30000

3.57	1.462	0.517	0.87	4.1	64.5
3.56	1.354	0.463	0.666	5.35	84.–

P10 fines 4/3 as above

1.53	1.436	0.254	0.411	3.72	58.5	~as expected
1.54	1.254	0.207	0.256	6.02	94.7	doesn't look good

Pellet cutting NEXT (see pg 80 for plan overview)

Pellet 3 & 4 dedicated to vertical & horiz. slicing

Pellets 6 & 7 ↓ ↓ ↓ ↓ ↓

from tangent saw cut edge: 1 mm slices are 0.055" w/ blade
1.2 .06

Low Density Vertical slices: 1 1.2 slices
6 1.0 slices } 7 altogether + end piece (marker) and polished end

Horizontal: 2 1.0 slices mid-section
1 0.5 top
1 1.0 bottom

High Density

VERTICAL: 5 slices (1 end lost to chipping) 1 end polished
~2mm ← 1 polished thick chunk
~1.2mm { 3 oxygenated
1 saved

horiz: 2 mid section
1 ea top & bottom (top chipped)

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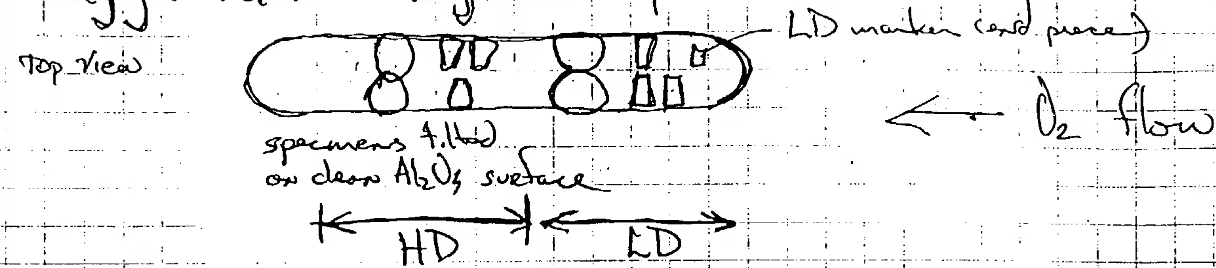
Date and ... entry. Have every possibly important entry wit... ...mit an Invention Disclosure of anything ... ...ly new and inventive.

IBM Technical Notebook

2/13/89

OXYGENATION DIAGRAM & scheme



2/14 in and to 600C @ 20C/min; 10C/min to 800C

15 min soak and start ramp to 600C @ 0.17°/min (10°/h)

To 600C @ 1:30 p.m. 2/15/89 in dry, CO_2-free O_2.

Pellet (1) O&C

1 mm vertical slices  10°/min to 850C, 10°/h to 600 2 day (48h), quench.

1) ✓ 1 slice for Jerry Burns (Raman)
2) ✓ 1 slice for Alex for XPS of fracture surface
3) save remainder for future use (dessicated)
2A) 1 xtra slice oxygenated

Pellet (2) O&C

1 mm horizontal slices 4 w/ outer slices discarded

1) 1 slice (1.0 mm) grind to ~0.5 mm & cut 3 3mm discs w/ ultrasonic in isopropyl alc (if possible).
   - 1 disc to Tom
   - 2 discs for TEM
   - 1 spare

2) 1 slice dedicated to ρ vs T to Dave's specs

Pellet (3) O&C

spare for ① x-ray lattice
② CO_2 evolution

The above understood ... Date ... and by ... Date

A1

Administrative Notes

2/16/89

C4 Synthesis Preparations/Notes (ref book IV, pg 46 & pg 14)

	oxide wt. frac.	atomic % Y, Ba, Cu	oxide M.W.
Y_2O_3 :	0.1751	.17	225.81
BaO :	0.4659	.33	153.34
CuO :	0.3625	.5	79.54

Go with the new

Example Calc: wt frac deriv.

				oxide wt frac
Y_2O_3	225.81 g × .17 m/mole = 38.39/2	= 19.19	19.19/109.56 =	0.1751
BaO	153.34 × .33	= 50.60	50.6/ =	0.4618(5)
CuO	79.54 × .5	= 39.77	39.77/ =	0.363
		109.56		0.9999

17.51 g Y_2O_3 1.287009

46.18 g BaO $\left\{\frac{197.35}{153.34} \times 46.18\right\}$ 59.43 g $BaCO_3$

Y_2O_3 19.19/.99 = 19.209 → 19.21 ⇒ × 1.5 28.81(5) 28.82

$BaCO_3$ 50.60(2) $\left(\frac{197.35}{153.34}\right)$ = 65.12(5) 97.69 97.69

CuO 39.77/.99 = ~~39.87~~ 59.71(5) 59.72

97.69 (0.777) = 75.91 − 97.69 =

186.23
− 21.78 CO_2
164.45

A2

Administrative Notes

FINAL Batch Size for REASONABLE BULK HANDLING

Y_2O_3 17.51/.999 = 17.52(7) ≈ 17.53

$BaCO_3$ 65.12(5)/.9999 = 65.12(5) ≈ 65.13 → 343.78

CuO 39.77/.999 = 39.80(9) ≈ 39.81

122.47

343.78(7) $BaCO_3$ Y_2O_3 17.53 weighed/transferred

tare 278.65

65.13 ✓ (-Δ 0.01(?))

382.~~76~~ CuO Mixing yield 122.29 g / 122.47 99.85% -Δ 0.15

t 343.78(7)

39.~~78~~ (-Δ 0.03)

151.42 OK

tare 1 87.49

63.93

140.48 X bad boat dimensions

tare 2 82.12

~~58.36~~ = 122.29 ✓

146.97(6)

88.61

58.36

sintered much more/some liq form

151.42

tare 1 144.37

7.05

87.55

less sintered, less liq

146.97

tare 2 141.74

5.23

88.64

- 12.28 CO_2 loss vs 14.53 expected (85% reacted) 2.25 to go

after GR I 108.71/110. = 98.8% -Δ 1.2%

197.3~~3~~ (.2)

88.64

108.69

	$Y_{\frac{1}{2}}O_3$	$BaCO_3$	CuO	SLS / Result
C4-1	0.17	0.33	0.5	assumed stoic pwdr
	0.16	0.36	0.5	analytical determination
C4-2	0.17	0.33	0.5	analytical liq
Δ	+0.01	-0.03	—	
C4-3	0.16	0.35	0.5	analytical
Δ	-0.01	+0.02	—	
Δnet	—	-0.01	—	
-3E	0.165	0.35	0.5	
Δ	-0.005	+0.02	—	
Δnet	+0.005	-0.01	—	
C4-4	0.16	0.34	0.5	analytical
Δ	—	-0.01	—	
Δnet	—	-0.02	—	
C4-5	0.16	0.34	0.49	analytical
Δ	—	—	-0.01	
Δnet	—	-0.02	-0.01	Species Implication → trace contam.
C4-6	0.16	0.34	0.47	$CuO \rightarrow Cu_2O$
Δ	—	—	-0.02	$BaCO_3 \rightarrow Ba(OH)_2$
Δnet	—	-0.02	-0.03	$Y_2O_3 \rightarrow Y_2O_3$ X
C4-7	0.16	0.34	0.478	
Δ	—	—	+0.008	
Δnet	—	-0.02	-0.022 ↑	

C4-7 TRANSFORM TO STOIC BASIS

Y_2O_3	$BaCO_3$	CuO	
0.17	0.31	~~0.478~~ 0.48	Correct for final batch calc

$\therefore$ Y_2O_3 is good as recieved

$BaCO_3$ is Barium rich by 0.02 at %

CuO is Copper rich by 0.02 at %

A3

Administrative Notes

Post Cal II ~60hs @ 950C in O_2 3/27

195.52 → post 88.64 (orig. rxn.)
88.64
106.88 − 108.69 = 1.81/2.25 = 80.5% of remainder

12.28
14.09/14.53 theor. = 97% + loss

Minimal liq. formation.

99.57g yield (due to contamination)

{ further contamination upon resubmersion to tube for cal III reduces yield further

Peaks @ ~30.2, 29.4, 28.5 2θ disturbing. May be $BaCO_3$, but could also be 2 ϕs

$n\lambda = 2d \sin\theta \Rightarrow d = \frac{\lambda}{2} \sin\theta$ 100 = 3.72

something wrong here

3.72 = ~~.7745 (sin θ)~~
4.803 = sin θ

4/19

FROM Plechaty : $Y_{.96}Ba_{2.16}Cu_3$ $(Y_{.32}Ba_{.72}Cu_1)O_{2.2}$

IN reference to VARIATIONAL study : 0.16 ±0.005 0.36 ±0.01 0.5 = 1.02

A4

Administrative Notes

A3

Administrative Notes

2/17/89 page 74 [redacted]975C "Property" Pellets f[redacted] co. variation study. - O_2

2g pellets should give enuf material for 4.4 mm thick sintered body allowing a slice to be cut w/ an interior & exterior surface.

7.5 g #1 stock 1.92 used for pellet (.17 .33 .5)

7.6 g #2 stock 1.90 ↓ ↓ (.15 .33 .52)

8.5 g #3 stock (.17 .35 .48)

9 #4

7.7 #5 to 600C @ 20°/min; 10°/min to 975C 4131-625C

3600/29,000 Quench 1h ~5:10

#1 P3

1.92	1.140	0.479	0.489	3.93	61.8
1.90	1.0	0.4	0.314	6.05	95—

#2 P3

1.91	1.126	0.490	0.488	3.91	61.5
1.88(?)	0.98	0.412	0.311	6.045	95—

#3	P2	1.133	0.503	0.507	3.85	60.5
	1.95	0.998	0.43	0.336	5.51	86.6
-Δ.1	1.85					

#4	P2	1.139	0.481	0.490	3.88	61—
	1.90	1.05	0.43	0.37	5.03	79—
	1.86					

#5	P3	1.145	0.46	0.474	3.99	62.7
	1.89	0.993	0.388	0.300	6.2	97.5
	1.86	~~1.05~~	~~0.41~~			

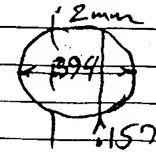
Administrative Notes 2/27

#1-5

Prep's slicing & oxygenation @ 600C for 66hrs in O_2

0.394 inches   0.157 in for center cut

.08 ~2 mm



#1 P-1   Pre 63.5%

Post 1.85   1.025   0.406   0.335   5.52   87

2/28 Property Pellet Summary (to date)

		stoic	liq	X	X	CO	
	t	#1	#2	#3	#4	#5	Run #
~~oxygenated~~	975	95	95	87	79	97.5	R3
in furnace 2/28	'950^{II}'	84	94	—	—	87	R2
	950	87	R done				R4
~~MICRO~~ STRUCTURES	ORIG 950	91	'97'	71	78	92	Dave's

950^{II} #1 & #5 pellets to temp @ 600C after 10°C/min Ramp @ 6:15 pm.
> oxygenation run

Out 10:00 A.M. 3/2 40h O_2

$5cc \times \frac{6.36g}{cc} \times = 31.8g$

$\frac{\pi (2.54)^2}{4} X = 5cc$

$5.07 \text{ cm}^2 X = 5cc$

$X = 5cm^3 / 5.07 cm^2$

$X \approx 1 cm$ or 1/2 inch - 1 inch with shrinkage

Administrative Notes

4/24/89

#1 - C3 wt% Cu 28.7

at% holes 36.0

Hole Concentration Conversion Formula:

Data: wt% Cu (total): 28.7

at% holes : 36.–

$$\frac{\text{at\% holes} - \text{at\% Cu}_{TOT}}{\text{Cu}_{TOT}} = \frac{36-28.7}{28.7} = 0.254$$

average 'over' valence

∴ add Cu valence (2) = 2.25 ≡ average valence Cu

2.25 (Cu_{TOTAL}) = 2.25 (3) = 6.75 total Cu val

from formula

$Y_1 Ba_2 Cu_3^{2.25}$

+ 7.00 total Ba + Y val

+2·2 +3

"

4

"

7

13.75

total charges

Take total charges & divide by two for O^{2-}

13.75/2 = 6.88 O atoms ⇒ $Y Ba_2 Cu_3^{2.25} O_{6.88}$

see page after next

Administrative Notes

Notes to Kristy concerning PELLET FORMING precalculations

To estimate ~~pellet~~ powder weight for pellet pressing:

A. take die dia & approx. hieght desired

1. calculate volume in cc. (i.e. $\frac{(1.22cm)^2 \times 0.35cm \times \pi}{4}$) = 0.41cc

B. Assume some reasonable 'green' density (unfired pressed pellet)
0.6-0.8 (60-80%) usual. for metals > 0.70 w/ small ave. part. dias. (i.e. 3 um).

Cu density theoretical

~~So~~ $(0.41\,cc / 0.8) \times 9.0\,g/cc \approx 3\,g$ of powder.

I pressed @ between 16,000 & 20,000 psi.
low side for pure metal ∴

$\frac{X}{(\text{dia die})^2}$ = desired pressure where X = 1" scale pressure

X ≈ 4,000 for 0.48" dia. die.

Administrative Notes K.V.f K.Kroll

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/9/89
SAMPLE Mosi-6
SOLVENT 150

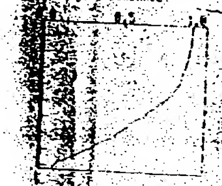
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	2.61(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 13 MIN 17 SEC

• DATA 0.95

TIME ABSORBANCE



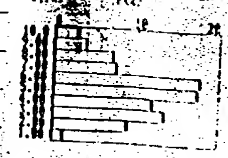
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	8.5	8.5
10.0-9.00	2.8	11.3
9.00-8.00	3.8	15.[illegible]
8.00-7.00	7.0	22.1
7.00-6.00	7.4	29.6
6.00-5.00	17.6	47.2
5.00-4.00	17.3	64.5
4.00-3.00	11.9	76.4
3.00-2.00	13.4	89.8
2.00-1.00	9.0	98.8
1.00-0.00	1.2	100.0

D(AVE) 4.84 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/14/89
SAMPLE $\alpha-Al_2O_3$
SOLVENT 150

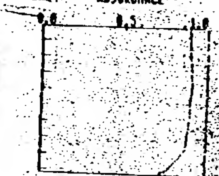
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	3.97(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 7 MIN 36 SEC

• DATA D~ 0.9

TIME ABSORBANCE



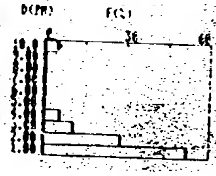
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0-9.0	4.5	4.5
9.00-8.00	0.0	4.5
8.00-7.00	0.0	4.5
7.00-6.00	0.0	4.5
6.00-5.00	0.0	4.5
5.00-4.00	0.0	4.5
4.00-3.00	5.5	10.0
3.00-2.00	10.5	20.6
2.00-1.00	[illegible].0	46.5
1.00-0.00	51.5	100.0

D(AVE) 0.97 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



5/1/89

Administrative Notes Analytical Results for C3 HD/LD STUDY - Holes

IBM RESEARCH CENTER ANALYTICAL LABORATORY

IBM

Request for Analysis

Use Ball Point Pen

REQUESTOR T. Shaw PROJECT NO REQUEST NO

DEPARTMENT LOCATION ROOM 25-225 PHONE

REQUESTOR'S SAMPLE IDENTIFICATION HD ox, LDox

APPROXIMATE COMPOSITION AND HISTORY OF SAMPLE YBaCu Oxide

ANALYSES REQUESTED

ANALYSIS METHOD

Cu

ANALYTICAL RESULTS

	HD	LD
Wt% "Holes"	33.5	34.2
→ Wt% Cu	(27.5)	(27.6)
	2.21	2.26
	6.81	6.89

tot # → %

\# Cu

After Reox: HDox 28.7 wt% Cu ← 28.6

purk @ 300C 36 holes

DATE SUBMITTED [illegible] DATE REPORTED 3/[illegible]/89 NOTEBOOK REFERENCE [illegible]

ANALYST [illegible] APPROVAL

Nº [illegible]

Administrative Notes

Notes to Kristy concerning Pellet forming precalculations

To estimate ~~pellet~~ powder weight for pellet pressing:

A. take die dia & approx. hieght desired

1. calculate volume in cc. $\left(\text{i.e. } \frac{(1.22\,cm)^2 \times 0.35\,cm \times \pi}{4}\right) = 0.41\,cc$

B. Assume some reasonable 'green' density (unfired pressed pellet)
0.6-0.8 (60-80%) usual. for metals > 0.70 w/ small ave. part. dias. (i.e. 3 μm).

$\left(0.41\,cc \,/\, 0.8\right) \times 9.0\,g/cc \approx 3\,g$ of powder.

C density theoretical

I pressed @ between 16,000 & 20,000 psi.
low side for pure metal ∴

$\frac{X}{(\text{dia die})^2}$ = desired pressure where X = 1" scale pressure

$X \approx 4{,}000$ for 0.48" dia. die.

Administrative Notes

4/24/89

#1 - Cu wt% Cu 28.7

wt% holes 36.0

Hole Concentration Conversion formula:

Data: wt% Cu (total): 28.7
wt% holes : 36.-

$$\frac{\text{wt\% holes} - \text{wt\% Cu}_{TOT}}{\text{Cu}_{TOT}} = \frac{36-28.7}{28.7} = 0.254$$

average 'over' valence

∴ add Cu valence (2) = 2.25 ≡ average valence Cu

2.25 (Cu$_{TOTAL}$) = 2.25 (3) = 6.75 total Cu val
+ 7.00 total Ba + Y val
13.75 total charges

from formula: $Y_1 Ba_2 Cu_3^{2.25}$

Ba+Y: +2·2 +3 = 4 + 3 = 7

Take total charges & divide by two for O^{2-}

13.75/2 = 6.88 O atoms ⇒ $Y Ba_2 Cu_3^{2.25} O_{6.88}$

see page after next

Administrative Notes

Notes to Kristy concerning Pellet Forming precalculations

To estimate ~~pellet~~ powder weight for pellet pressing:

A. take die dia. & approx. hieght desired

1. calculate volume in cc. $\left(\text{i.e. } \frac{(1.22\,cm)^2 \times 0.35\,cm \times \pi}{4}\right) = 0.41\,cc$

B. Assume some reasonable 'green' density (unfired pressed pellet) 0.6-0.8 (60-80%) usual. for metals > 0.70 w/ small ave. part. dias. (i.e. 3 um).

~~SO~~ $\left(0.41\,cc \,/\, 0.8\right) \times 9.0\,g/cc \approx 3\,g$ of powder.

C. density theoretical

I pressed @ between 16,000 & 20,000 psi.

low side for pure metal ∴

$\frac{X}{(\text{dia die})^2}$ = desired pressure where X = 1" scale pressure

$X \approx 4{,}000$ for 0.48" dia. die.

Administrative Notes

K. Kroll

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/9/89
SAMPLE ASCI-6
SOLVENT 150

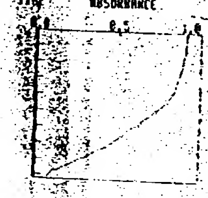
• CONDITIONS

SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 2.61(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 0.10(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 13 MIN 17 SEC

• DATA ∂.95

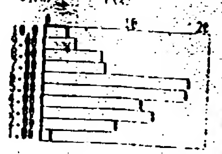


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.5	0.5
10.0- 9.00	2.8	11.3
9.00-8.00	3.8	15.1
8.00-7.00	7.0	22.1
7.00-6.00	7.4	29.6
6.00-5.00	17.6	47.2
5.00-4.00	17.3	64.5
4.00-3.00	11.9	76.4
3.00-2.00	13.4	89.8
2.00-1.00	9.0	98.8
1.00-0.00	1.2	100.0

D(AVE) 4.84 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/14/89
SAMPLE α-Al_2O_3
SOLVENT 150

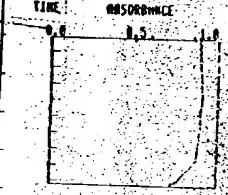
• CONDITIONS

SOLV.VISC 2.16(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 3.97(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 0.10(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 7 MIN 36 SEC

• DATA ∂~ 0.9

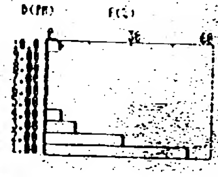


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	4.5	4.5
9.00-8.00	0.0	4.5
8.00-7.00	0.0	4.5
7.00-6.00	0.0	4.5
6.00-5.00	0.0	4.5
5.00-4.00	0.0	4.5
4.00-3.00	5.5	10.0
3.00-2.00	10.5	20.6
2.00-1.00	[illegible].0	46.5
1.00-0.00	51.5	100.0

D(AVE) 0.57 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



5/1/89

Administrative Notes Analytical Results for C3 HD/LD STUDY - Holes

IBM RESEARCH CENTER ANALYTICAL LABORATORY

IBM

Request for Analysis

Use Ball Point Pen

REQUESTOR T. Shaw PROJECT NO REQUEST NO

DEPARTMENT LOCATION ROOM 25-225 PHONE

REQUESTOR'S SAMPLE IDENTIFICATION HDox, LDox

APPROXIMATE COMPOSITION AND HISTORY OF SAMPLE YBaCu Oxide

ANALYSES REQUESTED

ANALYSIS METHOD

Cu

ANALYTICAL RESULTS

	HD	LD
wt% "Holes"	33.5	34.2
→ wt% Cu	27.5	27.0
	2.21	2.26
	6.81	6.89

~~tot # %~~

~~# Cu~~

After Reox: HDox 28.7 wt% Cu ← 23.6

purk @ 500C 36 holes

DATE SUBMITTED 3/.. /89 DATE REPORTED 3/24/89 NOTEBOOK REFERENCE .../121

ANALYST ... APPROVAL

Nº

5/2/07

Administrative Notes

Recalculation the HD, LD values w/ 28.7 % Cu

HD holes 33.5 ∴ $\frac{33.5-28.7}{28.7} = 0.167$

$2 + 0.167 = 2.167\ (3) = {}^{+}6.50$

$+\ 7$

$13.50 / 2 = 6.75 \Rightarrow Y Ba_2 Cu_3^{2.167} O_{6.75}$

LD $\frac{34.2-28.7}{28.7} = 0.192$ $2.192\ (3) = 6.58$

$+7$

$13.58 / 2 = 6.79$

∴ $Y Ba_2 Cu_3^{2.192} O_{6.79}$

with original anal. Cu values

HD $\frac{33.5-27.5}{27.5} = 0.22$ $2.22\ (3) = 6.66 + 7 = 13.66 / 2 = 6.83$

LD $\frac{34.2-27.0}{27} = 0.27$ $2.27\ (3) = 6.81 + 7 = 13.81 / 2 = 6.90(5)$

w/ ave. 27.5 + 27 = 27.25

LD $\frac{34.2-27.25}{27.25} = 0.25(5)$ $2.255\ (3) = 6.765 = 13.765 / 2 = 6.88$

HD $\frac{33.5-27.25}{27.25} = 0.23$ $2.23\ (3) = 6.69 \cancel{6.845}$ Co

Composition #	Rel Pellet density (%)*	Actual density	Green Rel δ	Green act δ
1	84 (90.8)	5.34	62.9	4.0
2	98.9	5.95	61.5	3.90
3	71	4.52	60	3.82
4	77.8	4.95	62	3.93
5	87 (91)	5.54	63.9	4.03

* after 1h sinter @ 950C